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Preliminary Operation of the Fluidized-Bed Combustion Facility for Hazardous Waste Disposal Research: Test Results and Evaluation

Acurex Corporation
Research Triangle Park, NC

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PRELIMINARY OPERATION OF THE FLUIDIZED-BED COMBUSTION FACILITY FOR HAZARDOUS WASTE DISPOSAL RESEARCH

Test Results and Evaluation

By

R. Clayton, H. Dempsey, and R. Machilek
Acurex Corporation
Energy and Environmental Division
P. O. Box 13109
Research Triangle Park, NC 27709

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EPA Project Officer
John H. Wasser

Air and Energy Engineering Research Laboratory
Office of Environmental Engineering and Technology
Research Triangle Park, NC 27711

AIR AND ENERGY ENGINEERING RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

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16. ABSTRACT The report describes Phase I of a program to provide research data on the destruction of hazardous wastes in a fluidized-bed combustor (FBC). The report addresses three primary areas of the program: facility operation, sampling and analysis, and health and safety. The FBC was operated at a nominal temperature of 982 C for each test run. The bed height changed somewhat from run to run, but the residence time at a temperature of about 982 C was maintained at about 0.8 sec. CO and CO2 concentrations in the freeboard were monitored continuously, along with downstream O2 and total hydrocarbon levels. Operating parameters were maintained easily, but were less flexible than desired. Phase II of this program will re-evaluate and continue the research effort, based on the Phase I data and conclusions.

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Wastes	Stationary Sources	14G
Waste Treatment	Hazardous Waste	
Toxicity	Waste Disposal	06T
Combustion		21B
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ABSTRACT

The first phase of a program designed to provide research data on the destruction of hazardous wastes in a fluidized bed combustor was performed for the Environmental Protection Agency's Industrial Processes Branch.* The basic program was developed and conducted by Acurex Corporation with the sampling and analytical functions provided by EPA's Technical Support Staff (TSS)** The health and safety planning and protocol development was the responsibility of Acurex Corporation with the support of TRW, Inc. for the assemblage of a safety training seminar and monitoring of safety-related functions during testing. This final report addresses three primary areas of the program:

1) Facility Operation
2) Sampling and Analysis
3) Health and Safety

The Facility Operations section documents the engineering aspects of facility preparation, start-up, test burns, and shut down. Each section in turn addresses analogous phases of the study. The sampling and analysis results are presented by EPA-TSS while the health and safety observations and conclusions are provided by TRW, Inc.

The fluidized bed combustor was operated at a nominal temperature of 1800°F for each test run. The bed height changed somewhat from run to run but the residence time at a temperature of ~1800°F was maintained at ~.8 second. Carbon monoxide and carbon dioxide concentrations in the freeboard were continuously monitored along with downstream oxygen and total hydrocarbon levels.

Operating parameters were found to be easily maintained but less flexible than desired. Phase II of this program will re-evaluate and continue the research effort based on the data and conclusions of Phase I.

The monitoring of test activities with respect to personnel health and safety led to the evolution of documented protocols and discovery of new problem areas, particularly facility limitations to handling bonafide hazardous wastes. No adverse health effects were experienced during the test series, but several minor spills of fuel/waste mixture provided valuable insight into the difficulty of clean-up. Another result from this study was an empirically-derived cost of such a research effort attributed to health and safety concerns.

(*) Now EPA/AEERL's Hazardous Air Technology Branch.
(**) Now EPA/AEERL's Technical Support Office.

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(*) EPA's Industrial Environmental Research Laboratory (IERL) at Research Triangle Park, NC, is now EPA's Air and Energy Engineering Research Laboratory (AEERL).

SUMMARY

As part of an interagency coordinated research program to provide technical support for EPA's hazardous waste permitting activities, AEERL's Industrial Processes Branch sponsored a program to incinerate surrogate wastes in a fluidized bed combustor (FBC). A multi-phased program was developed to utilize the FBC for a variety of hazardous waste incineration studies including destruction and removal efficiency (DRE) testing, sampling and analysis methods evaluation and development, and incineration and control technology development. The existing pilot-scale atmospheric fluidized bed combustion facility, located in EPA's Environmental Research Center in Research Triangle Park, NC, was utilized for this study.

Phase I of the program was designed to (1) develop, test and evaluate facility operation protocols with emphasis on facility safety and personnel health, (2) determine destruction and removal efficiencies for selected test compounds at a given set of operating conditions, and (3) test selected state-of-the-art sampling trains and analysis methods.

The six month Phase I program included the development of a comprehensive Test Plan including QA/QC and Health and Safety Plans for facility operation and sampling and analysis activities, modification of the FBC to accomplish the goals of the plan, and a three-week test burn for data collection. A post-test evaluation period was designed to analyze the results and re-appraise the program's goals and direction.

Test compounds were chosen to represent a full range of volatility and ease of destruction which would allow the testing of three sampling and analysis methods including: (1) Volatile Organic Sample Train (VOST) for measuring the volatile constituents, (2) Source Assessment Sampling System with a glass XAD-2

Resin module (Glass SASS) for collecting the semi-volatiles, and (3) Stack Dilution Sampling System (SDSS) for measuring the condensible organic components in the flue gas. Test compounds were chosen which were not listed as principle organic hazardous constituents in Appendix VIII of the Resource Conservation and Recovery Act (RCRA).

These surrogate wastes were mixed with No. 2 fuel oil at 1 to 2 percent by weight concentrations to be injected into the fluidized bed.

Facility modifications included the following:

1. Exhaust stack extension to ensure adequate dispersion of any flue gas pollutants.
2. Installation of a gas-fired burner in the combustor for bed pre-heat and fuel oil ignition.
3. Installation of a liquid fuel feed system to safely handle and inject the fuel/waste mixtures.
4. Recasting of combustor section to a circular cross-section with more abrasive-resistant material.
5. Modification of particulate clean-up devices (cyclone, baghouse) to safely catch and dispose of potentially hazardous residues.
6. Addition and modification of facility safety controls.

A typical test run required approximately 24 hours beginning 12 to 18 hours before data collection began. The facility draft fan, blower, electric and gas preheater were operated to preheat the combustor, bed material and downstream facility components.

After a clean oil start-up burn time of 3 to 6 hours, the facility was operated at a nominal bed temperature of 1800°F for each fuel/waste burn. Apparent residence time at temperature was approximately 0.8 second. The bed was approximately 25 inches (slumped) of .020 to .050-inch spherical bauxite beads expanded to 30-35 inches. After data collection was completed, the facility was operated on clean fuel for approximately 30 minutes before fuel shut-off and facility cooldown.

Two fuel/waste mixtures were burned - one containing the non-volatile and semi-volatile compounds and the other containing the volatile compounds. The Glass SASS and SDSS methods were conducted simultaneously on three separate test dates. The three VOST runs were made successively during one test day.

The data provided DRE's for the six compounds in the 99.99% range, verifying the usefulness of the facility for hazardous waste research. Phase I of the program was designed to provide not only quantitative data but also operational data to evaluate the protocols developed for facility sampling and analysis operations with particular emphasis on QA/QC, personnel health and safety, and program goals.

In order to develop and evaluate a comprehensive health and safety plan, a third-party was utilized to assist in the development of protocols, training of personnel, and evaluation of performance and procedures.

The facility was found to perform satisfactorily with one problem affecting the test. The fuel feed nozzle plugged during operation causing a premature shutdown. All goals of the facility operation plans were met by performing the required test burns and providing the data necessary to evaluate the facility's capabilities and plan future testing.

The goals of the sampling and analysis functions of the program were also achieved. The three sampling methods were tested and analytical determinations of the data were made to provide the DRE determinations. The operational experience with each sampling method provided the data to evaluate the methods and analytical procedures and determine the logistics necessary for data collection and analysis for hazardous waste studies using the FBC facility.

The health and safety aspects of the program were a primary focal point of the test plans to achieve the facility operation and sampling and analysis goals. The accomplishment of those goals provided the opportunity to evaluate the health and safety protocols developed for the program and the adherence to the protocols by the facility operations and sampling crews. A determination of health and safety-related costs was made as part of this effort.

Section 4 of this report outlines the specific conclusions and recommendations relating to each program evaluation area.

The facility modifications and operation was the responsibility of Acurex Corporation. Sampling methods testing and data analyses were performed by AEERL's Technical Support Staff. The health and safety support and evaluation was provided by TRW, Inc.

SECTION 1

INTRODUCTION

The Environmental Protection Agency's Industrial Processes Branch sponsored a program to use an existing in-house fluidized bed combustion facility (FBC) to gather data in support of permitting requirements for hazardous waste disposal as charged by the Resource Conservation and Recovery Act of 1976 (RCRA). A multi-phased program was developed to utilize the FBC for disposal and removal efficiency (DRE) testing, sampling and analysis methods development, and incineration and control technology development. The first phase of this program focused on (1) development, testing and refinement of operating protocols for the facility, (2) establishing DRE's for selected surrogate hazardous wastes and (3) testing of selected state-of-the-art sampling and analysis trains. A post test evaluation period was designed to analyze the results and observations and provide direction for specific goals of succeeding phases of the program.

To meet the above goals, an experimental program was developed by Acurex Corporation, Research Triangle Park, North Carolina with support from EPA's Technical Support Staff and direction from the Industrial Processes Branch. The program involved the incineration of fuel oil spiked with organic chemical compounds, representing a full range of volatility and ease of destruction, at a single set of operating conditions. Flue gas sampling was accomplished by utilizing three sampling methods, each designed for detecting the test compounds within a specific volatility range.

An overview of the program is found in the Work Plan for Work Assignment 44, EPA Contract 68-02-3176. Specific Phase I goals, tasks, and direction are described in the FBC Test Plan included in this report.

This report documents all aspects of the program as performed. It includes (1) the facility description, operation, and results authored by Acurex Corporation who was also responsible for organizing the report; 2) sampling and analysis methods and findings of the flue gas sampling activities conducted and documented by EPA's Technical Support Staff (Sections 3, 4.2, 6.2); 3) and documentation and evaluation by TRW, Inc. of the health and safety protocols developed for use in this test program (Sections 5, 6.3).

SECTION 2

FACILITY OPERATION

In order to maintain reasonable brevity, this report will concentrate on deviations from the Test Plan of this report. Brief descriptions of the facility and operation will be included but reference should be made to the Test Plan for detailed descriptions and explanations.

2.1 TEST FACILITY

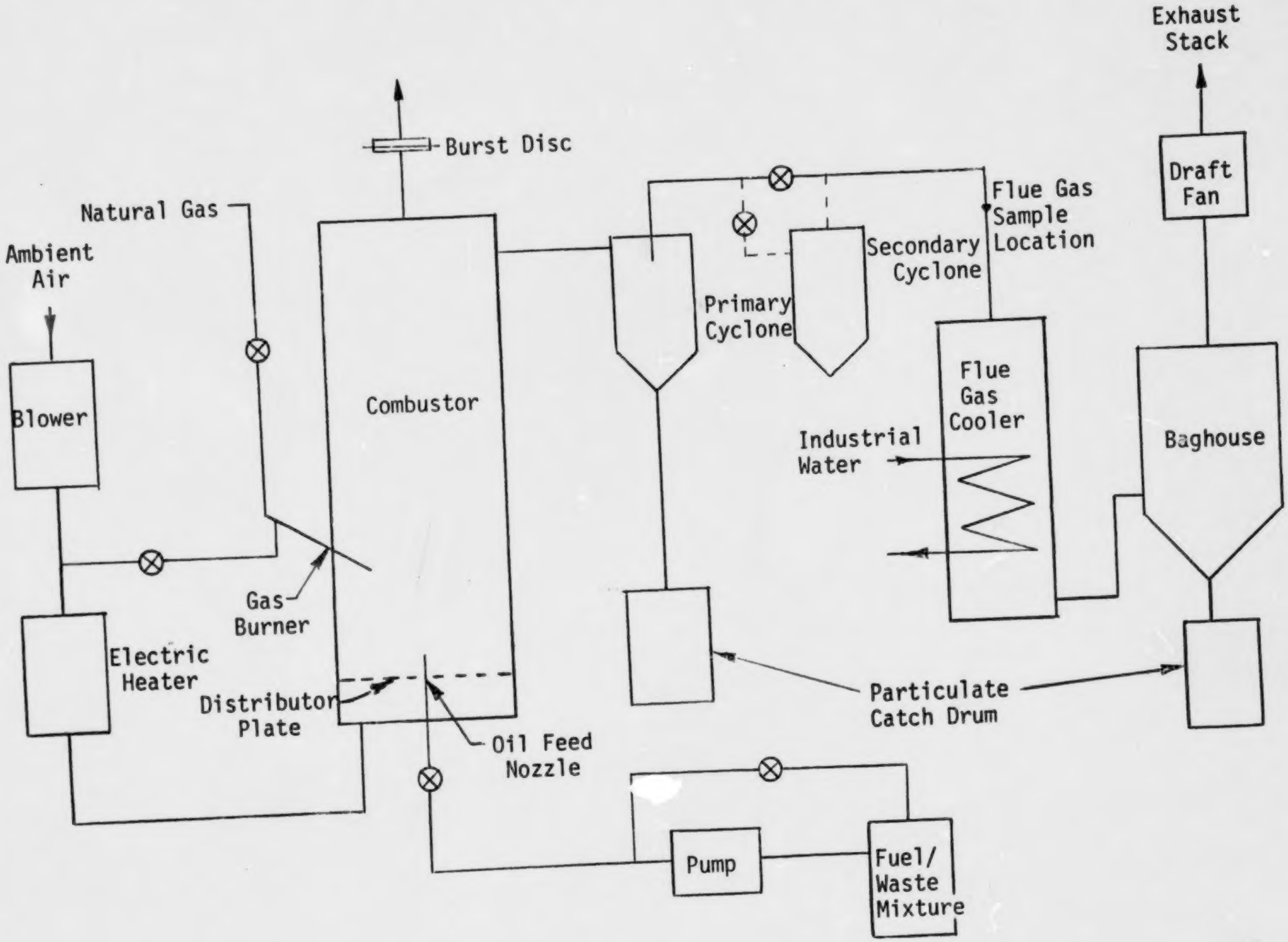
The original FBC facility was designed to be a versatile coal burning pilot plant with various options for combustion and flue gas clean up studies. Figure 2-1 shows a schematic of the facility as used for Phase I.

Several modifications were required on the facility and described in the Test Plan. No major deviations from the plan were made although several minor revisions evolved during the construction and testing phases. The modifications are described below.

2.1.1 Combustor Section

The combustor section refractory was recast to a 15-inch internal diameter. Babcock and Wilcox's Kao-Phos 93 was used as planned for the inner material but was not cast to the outer shell. A 2-inch layer of B&W's 'M-board', an insulating ceramic board, was fixed to the outer shell. Next, an insulating refractory, Kao-Lite 2300 was applied from the "M"-board to a 21" I.D.

A 3-inch thickness of Kao-Phos 93 was then cast to the desired internal diameter. This multi-layer lining was required to maintain sufficiently low skin temperatures and combustor weight. Figure 2-2 shows cross-section of the combustor with the port locations shown.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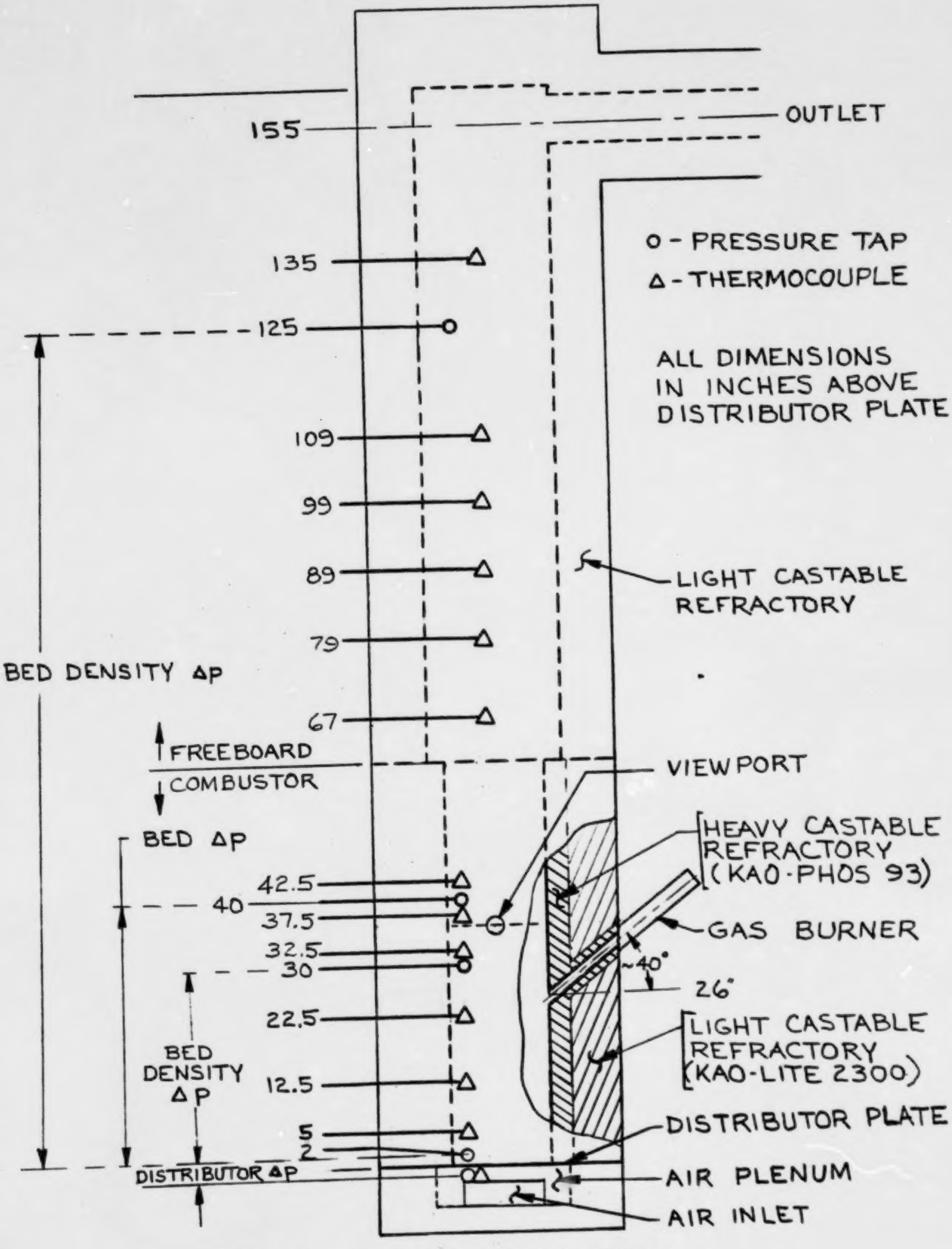
Natural Gas
Ambient Air
Blower
Combustor
Gas Burner
Electric Heater
Distributor Plate
Oil Feed Nozzle
Primary Cyclone
Secondary Cyclone
Flue Gas Sample Location
Flue Gas Cooler
Industrial Water
Baghouse
Particulate Catch Drum
Pump
Fuel/ Waste Mixture

Figure 2-1. FBC Facility Schematic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OUTLET
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125
O - PRESSURE TAP
Δ - THERMOCOUPLE
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99
89
79
67
LIGHT CASTABLE REFRACTORY
BED DENSITY ΔP
FREEBOARD
COMBUSTOR
VIEWPORT
BED ΔP
HEAVY CASTABLE REFRACTORY (KAO-PHOS 93)
42.5
40
37.5
GAS BURNER
32.5
30
~40°
26°
22.5
BED DENSITY ΔP
12.5
LIGHT CASTABLE REFRACTORY (KAO-LITE 2300)
5
2
DISTRIBUTOR PLATE
DISTRIBUTOR ΔP
AIR PLENUM
AIR INLET

Figure 2-2. FBC Combustor-Port Locations.

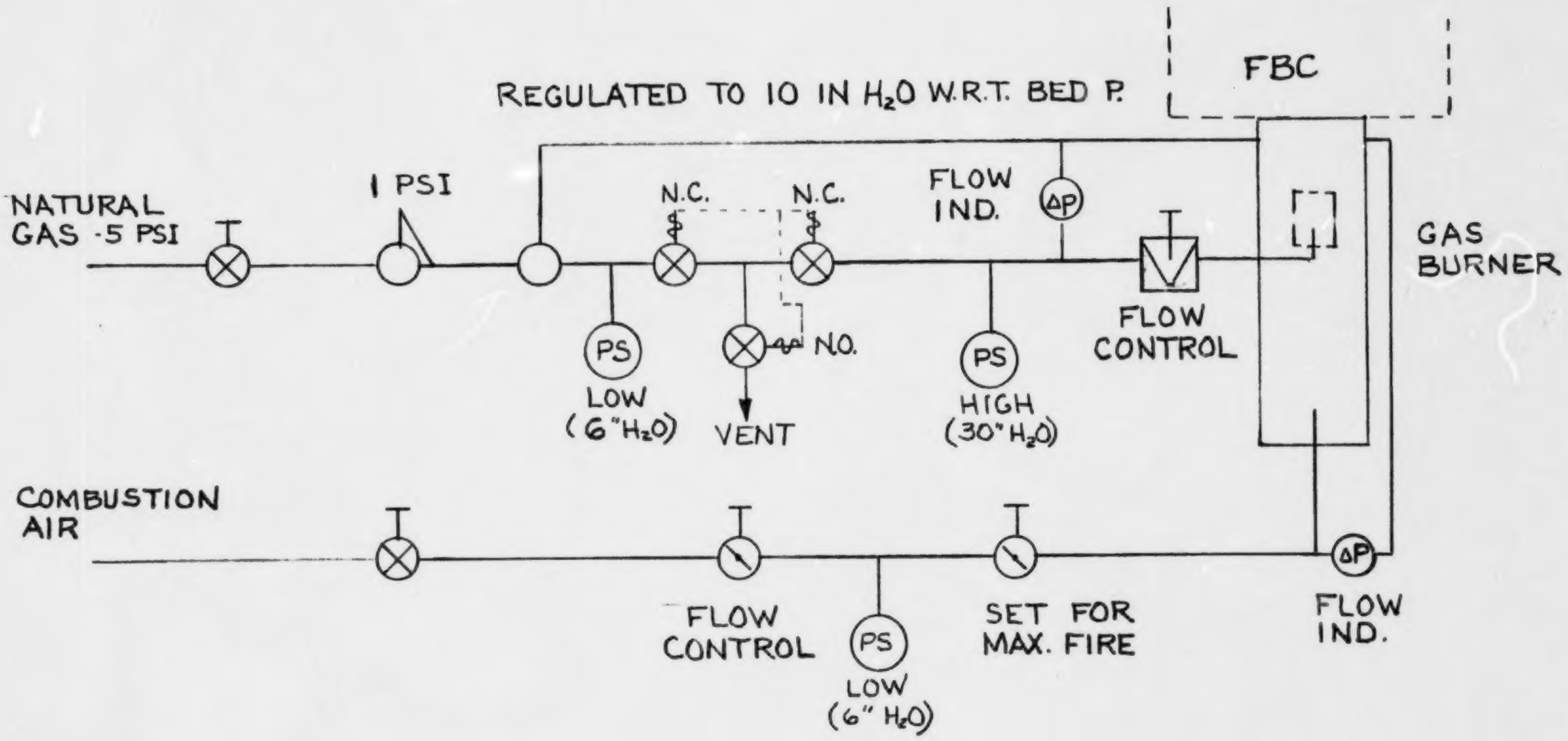
2.1.2 Gas Burner

An Eclipse model 51.5 gas burner was installed in the combustor section through an existing cover plate for a cooling tube bundle location. Figure 2-2 shows the location and construction of the port. The burner was threaded into a half-coupling welded into the cover plate at a 40° angle to horizontal, allowing the flame to impinge on the slumped bed but preventing the collection and retention of bed material in the burner during operation. The piping and instrumentation schematic is shown in Figure 2-3. The safety system is provided by a Protectofier Combustion Safeguard and meets all requirements of the National Fire Protection Association Standards.

2.1.3 Liquid Fuel Feed System

The fuel system built to supply the fuel/waste mixture to the combustor is basically as designed and described in the Test Plan. Modifications to the plan include the following:

- Flow measurement was provided by a single rotometer between the 3-way manual valve and the fuel shut-off valve. It was determined that a flow totalizer capable of providing highly accurate data was not necessary or cost-effective for Phase I.
- Felt-cartridge oil filters were installed in the flow control module for both the clean and spiked oil systems.
- One fuel shutoff valve was deemed sufficient after it was decided to run on oil only when operators were present. Similarly, the high- low pressure switch in the flow control module was determined to be unnecessary, but high pressure switches were installed in the pump module to shut off the pumps in case of an over-pressure condition.
- Pressures in the pumping loops were controlled by an oil pressure regulator in the supply line to the combustor and a recirculation loop oil pressure relief valve.


REGULATED TO 10 IN H_2O W.R.T. BED P.
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GAS BURNER
PS
LOW (6" H_2O)
N.O.
VENT
PS
HIGH (30" H_2O)
FLOW CONTROL
COMBUSTION AIR
FLOW CONTROL
PS
LOW (6" H_2O)
SET FOR MAX. FIRE
ΔP
FLOW IND.

Figure 2-3. Gas Burner - P & ID

- Straight runs of copper tubing between the fuel shed and flow control module were given mechanical protection by rigid polyvinylchloride (PVC) pipe with clear plastic tubing clamped to the pipe where the fuel tubing was bent. The clear tubing is located where damage to the copper is unlikely.

A schematic of the fuel feed system is shown in figure 2-4 with the fuel handling shed depicted in Figure 2-5.

Two oil feed nozzles were used during the test. Both used the same injection design but differed in the cooling jacket. The first nozzle, as shown in Figure 2-6, had a separate 1/4" oil feed tube which slipped through a cooling tube jacket. This design would allow the flexibility of changing the nozzle design without building another cooling jacket, but had the disadvantage of lower cooling capacity.

The second feed nozzle, built after the first cooling jacket developed leaks, was an integral unit in which the oil feed tube was also the inner wall of the cooling jacket. The cooling was also enhanced by the increased size (3/8" of the cooling water inlet and outlet stubs. Figure 2-7 depicts the second feed nozzle.

All electrical equipment and field wiring in the fuel feed shed was chosen for hazardous location use since volatile flammable compounds would be handled in the shed.

2.1.4 Flue Gas Clean Up System

The particulate catch collection system was designed to provide a quick and safe method of changing drums and disposing of the residue. Drum lids were modified to attach to the clean up device downchutes with quick-connect hose ends. By disconnecting drums while the draft fan is on and capping the openings, fugitive emissions will be negligible. The modified lids will be replaced by the original lids for residue disposal. Drums are transported

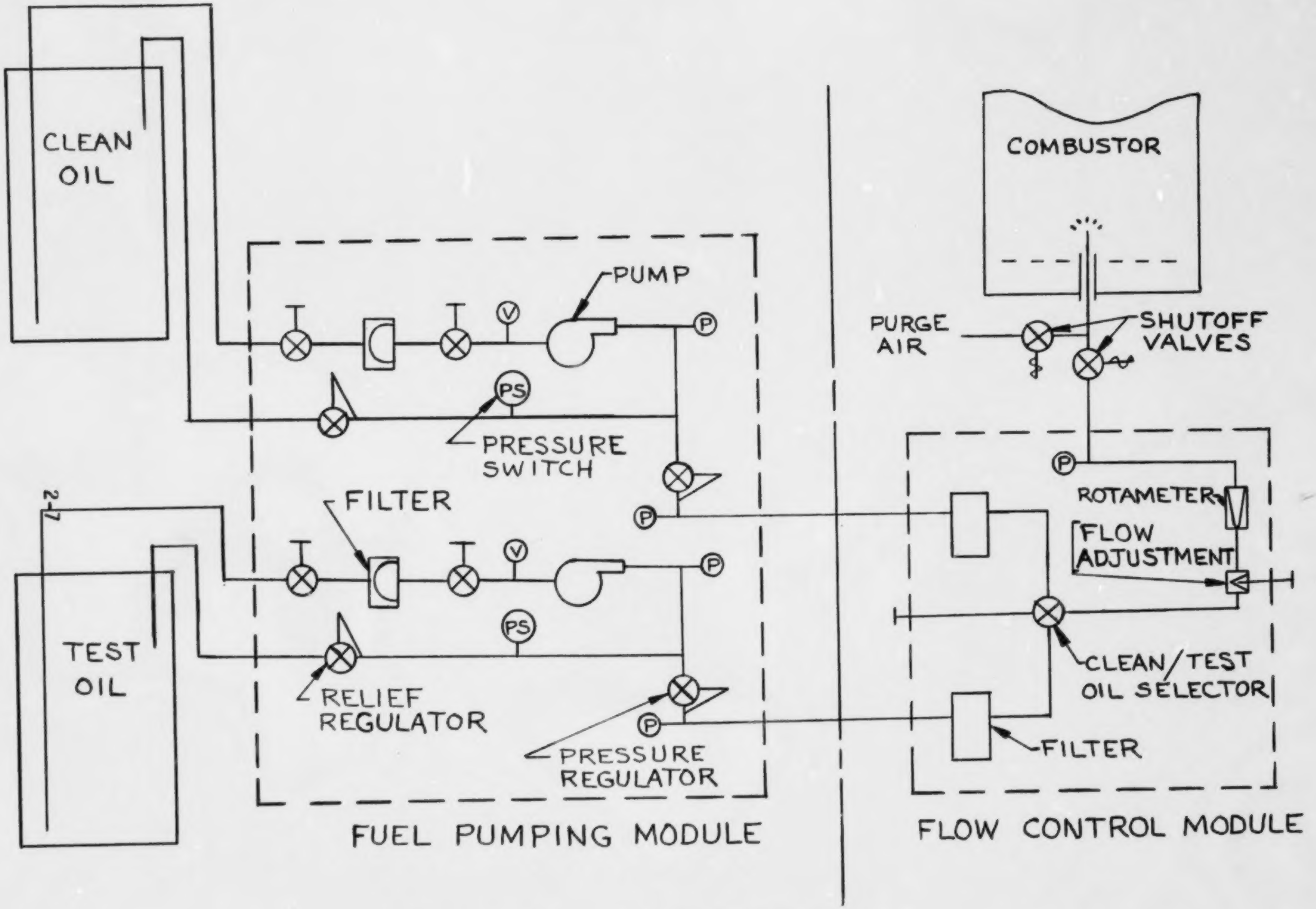
CLEAN OIL
TEST OIL
PUMP
PRESSURE SWITCH
FILTER
RELIEF REGULATOR
PRESSURE REGULATOR
FUEL PUMPING MODULE
COMBUSTOR
PURGE AIR
SHUTOFF VALVES
ROTAMETER
FLOW ADJUSTMENT
CLEAN/TEST OIL SELECTOR
FILTER
FLOW CONTROL MODULE
P
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Figure 2-4. FBC Liquid Fuel Feed System.

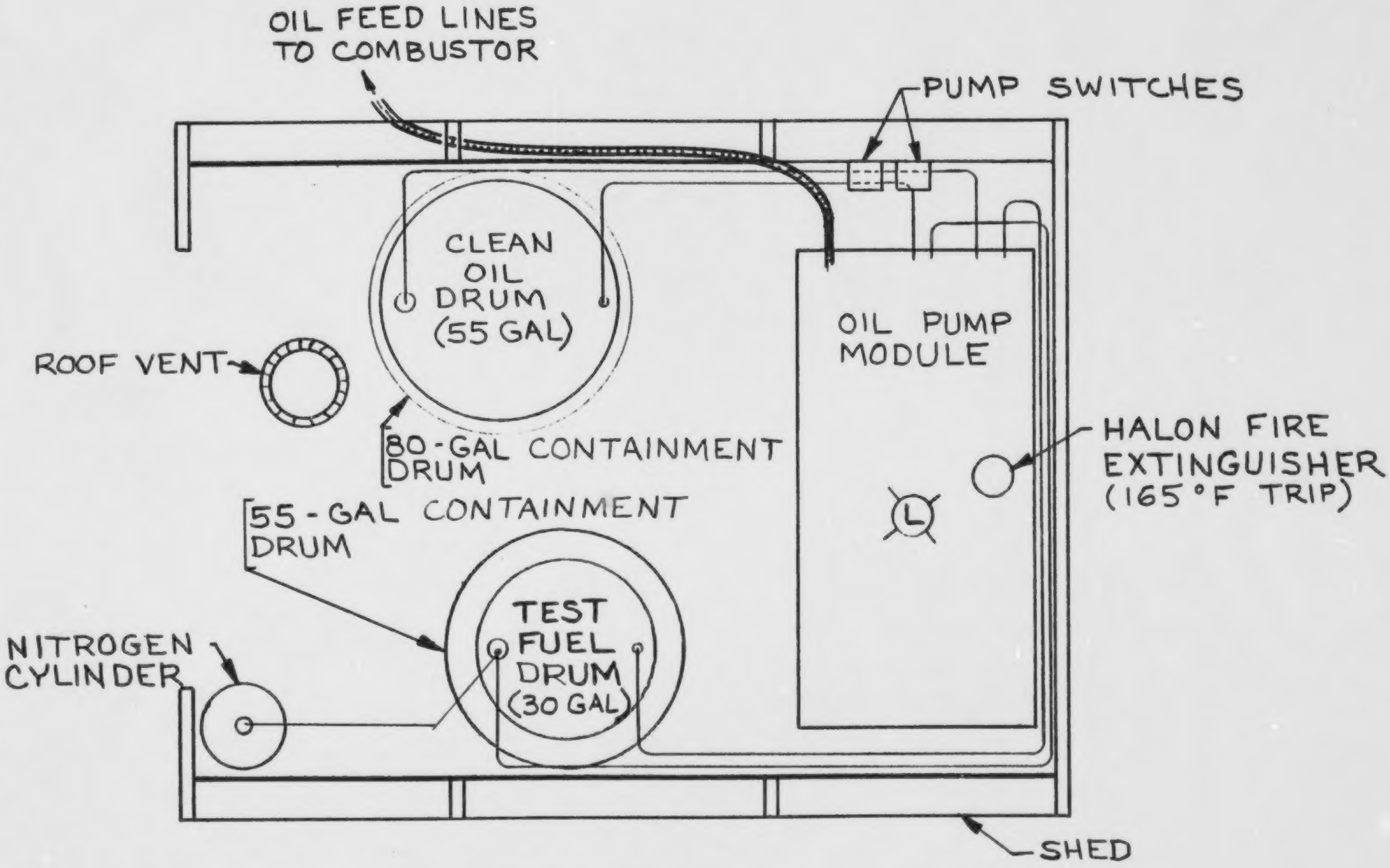
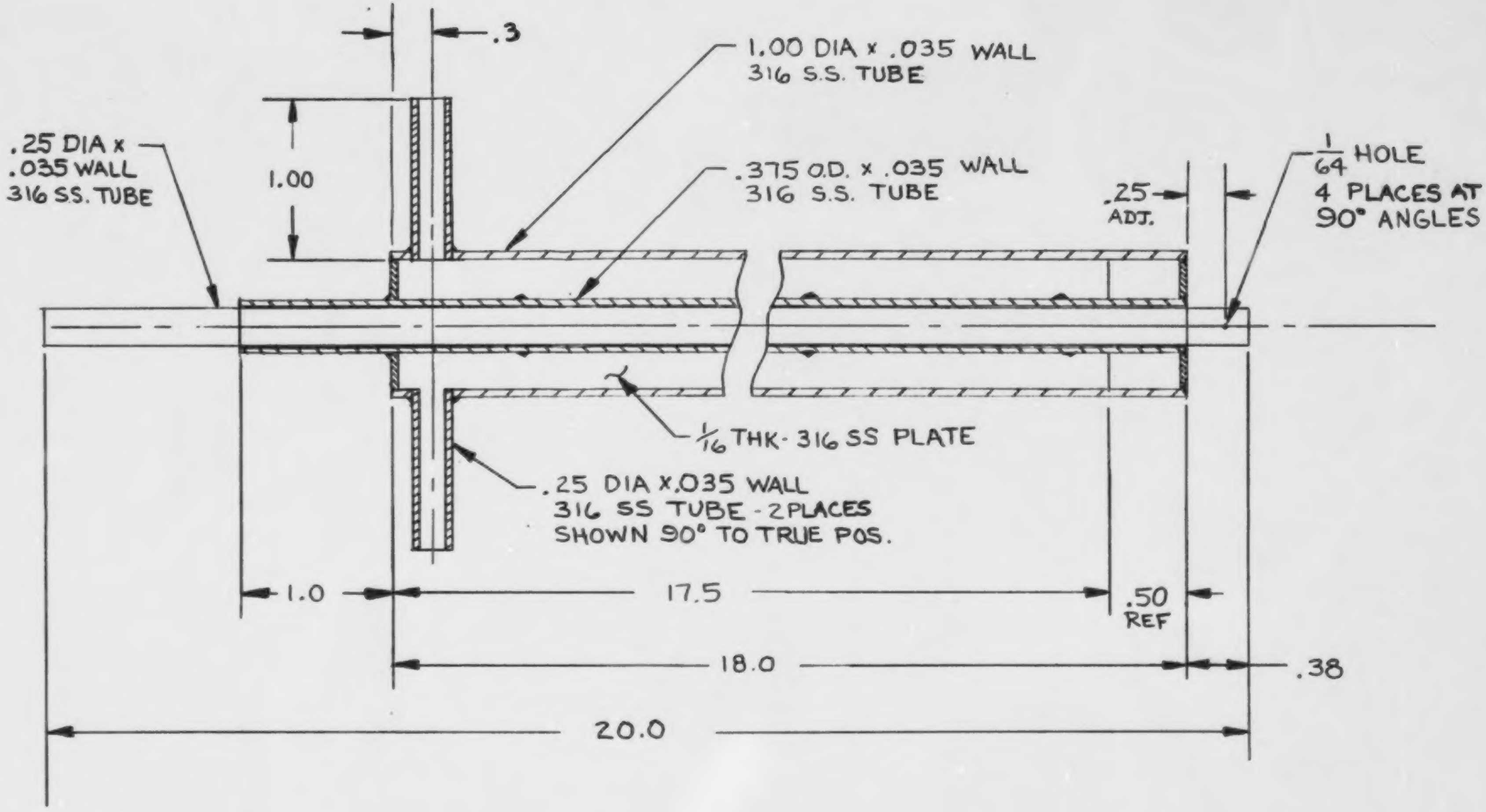
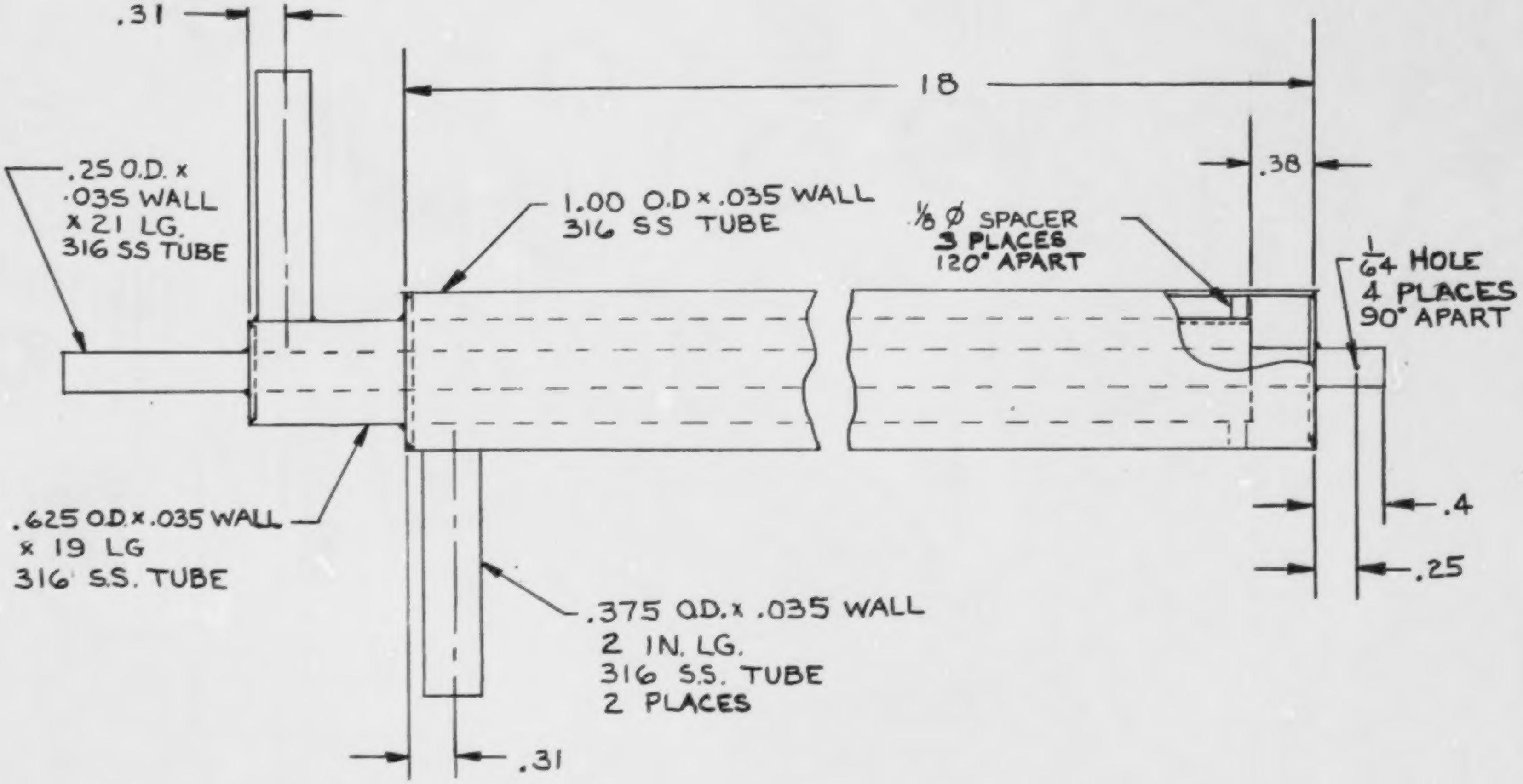

OIL FEED LINES TO COMBUSTOR
PUMP SWITCHES
CLEAN OIL DRUM (55 GAL)
OIL PUMP MODULE
ROOF VENT
80-GAL CONTAINMENT DRUM
HALON FIRE EXTINGUISHER (165 °F TRIP)
L
55-GAL CONTAINMENT DRUM
TEST FUEL DRUM (30 GAL)
NITROGEN CYLINDER
SHED

Figure 2-5. Liquid Fuel Handling Shed.


.3
1.00 DIA x .035 WALL
316 S.S. TUBE
.25 DIA x
.035 WALL
316 SS. TUBE
1.00
.375 O.D. x .035 WALL
316 S.S. TUBE
.25
ADJ.
$\frac{1}{64}$ HOLE
4 PLACES AT
90° ANGLES
$\frac{1}{16}$ THK- 316 SS PLATE
.25 DIA x .035 WALL
316 SS TUBE - 2 PLACES
SHOWN 90° TO TRUE POS.
1.0
17.5
.50
REF
18.0
.38
20.0

ALL DIMS. IN INCHES

Figure 2-6. Oil Feed Nozzle - I (2-piece).


.31
18
.38
.25 O.D. x
.035 WALL
x 21 LG.
316 SS TUBE
1.00 O.D x .035 WALL
316 SS TUBE
1/8 Ø SPACER
3 PLACES
120° APART
1/64 HOLE
4 PLACES
90° APART
.4
.25
.625 O.D. x .035 WALL
x 19 LG
316 S.S. TUBE
.375 O.D. x .035 WALL
2 IN. LG.
316 S.S. TUBE
2 PLACES
.31

ALL DIMS. IN INCHES

Figure 2-7. Oil Feed Nozzle -II (1-piece).

within the facility and raised into position by a manually operated 3-wheel drum lifter.

2.1.5 Exhaust Stack

Figure 2-8 shows the exhaust stack modification made on the facility. The existing draft fan was straightened with respect to the building wall and a new stand built and installed. An eight-foot stack extension with 2-foot rain cap was fabricated to the existing stack and guyed to the wall with two rigid poles. The stack was extended to ensure adequate dispersion of the exhaust gases if meteorological conditions favored stack or building downwash.

2.1.6 Safety System

The facility safety system was modified to provide a prompt and safe shutdown of critical components in case of a potentially dangerous operational upset condition such as draft fan or blower shutdown. Table 2-1 indicates the conditions detected and responses given by the safety devices. Operator responses are noted as well.

2.1.7 Sampling System

The combustion process was continuously monitored by measuring flue gas CO and CO_2 concentrations in the freeboard. The sample was extracted through a water-cooled stainless steel probe and transported through a teflon line to the analyzer rack. Data were continuously recorded on an Esterline Angus chart recorder. Since the O_2, NO_x and THC concentrations were not required, those analyzers were not operated during Phase I due to the potential for contaminating the instruments. Downstream oxygen and hydrocarbon concentrations were monitored by a Teledyne analyzer and alarm system.

The sampling system is shown in Figure 2-9. Complete instrument specifications are provided in the Phase I QA/QC Project Plan.

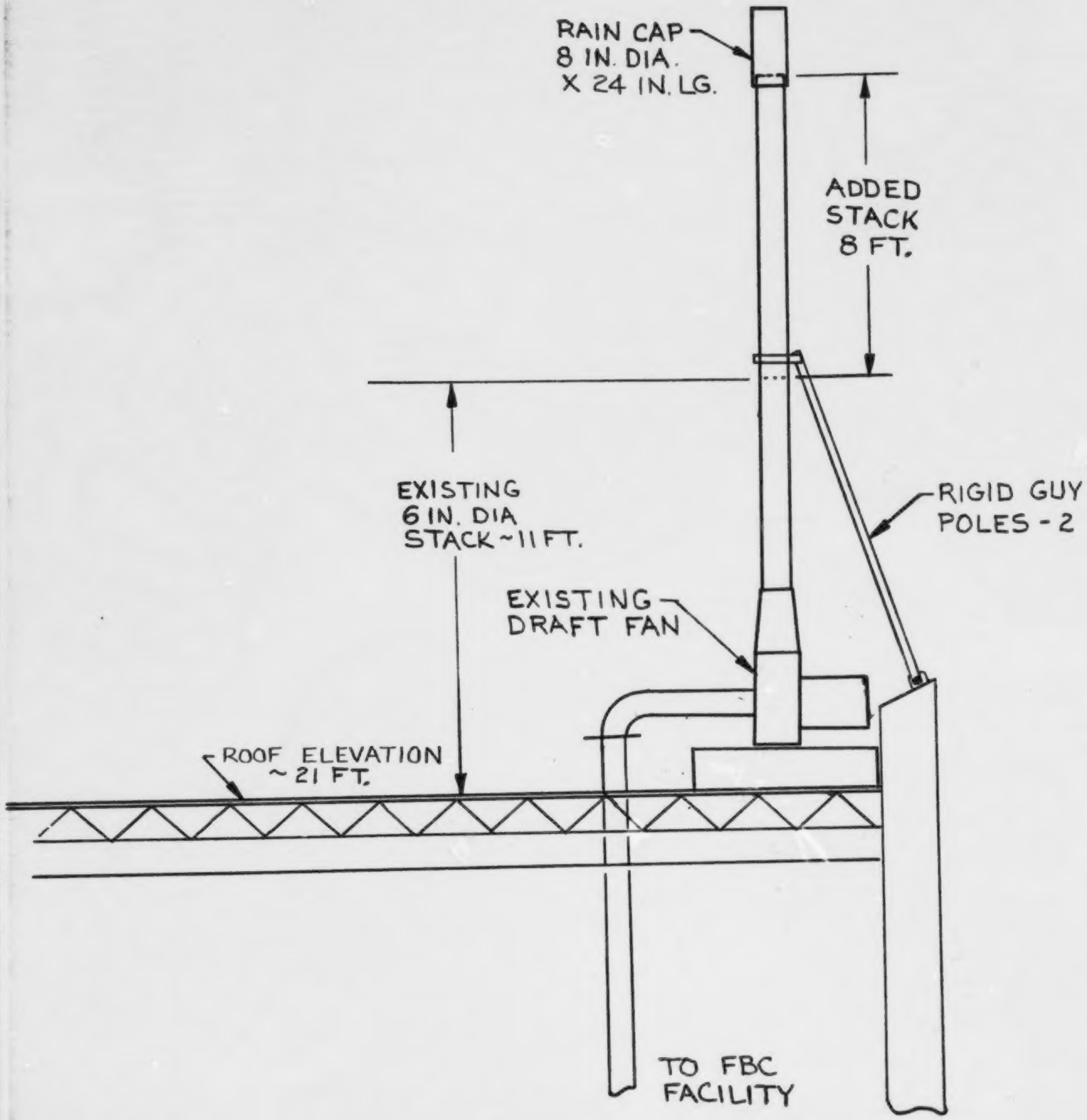

RAIN CAP
8 IN. DIA.
X 24 IN. LG.
ADDED
STACK
8 FT.
EXISTING
6 IN. DIA
STACK ~11 FT.
RIGID GUY
POLES - 2
EXISTING
DRAFT FAN
ROOF ELEVATION
~21 FT.
TO FBC
FACILITY

Figure 2-8. FBC Exhaust Stack.

TABLE 2-1 . SAFETY ALARMS AND INTERLOCKS

Condition	Limit	Device	Response
High Blower Inlet Temp.	150°F	Temp Switch	Power Off, Heater Off, Fuel Off
Low Air Velocity	15 scfm	Δp Switch	Heater Off, Fuel Off
No Flame	-	UV Detector	Gas, Oil Fuel Off
Low Draft	-10 in H_2O	Pressure Switch	Heater Off, Fuel Off
High Bed Temperature	2500°F	Temp Switch	Fuel Off
High Baghouse Δp	10 in H_2O	Δp Switch	Light - Operator to verify cleaning
Low Water Pressure Combustor/Cooler	40 psi	Δp Switch	Alarm - Operator to monitor & evaluate to prevent baghouse overtemp
Low O_2	5%	Teledyne Analyzer	Alarm - Operator to reduce fuel or increase air
High Combustibles	1%	Teledyne Analyzer	Alarm - Operator to reduce fuel or increase air

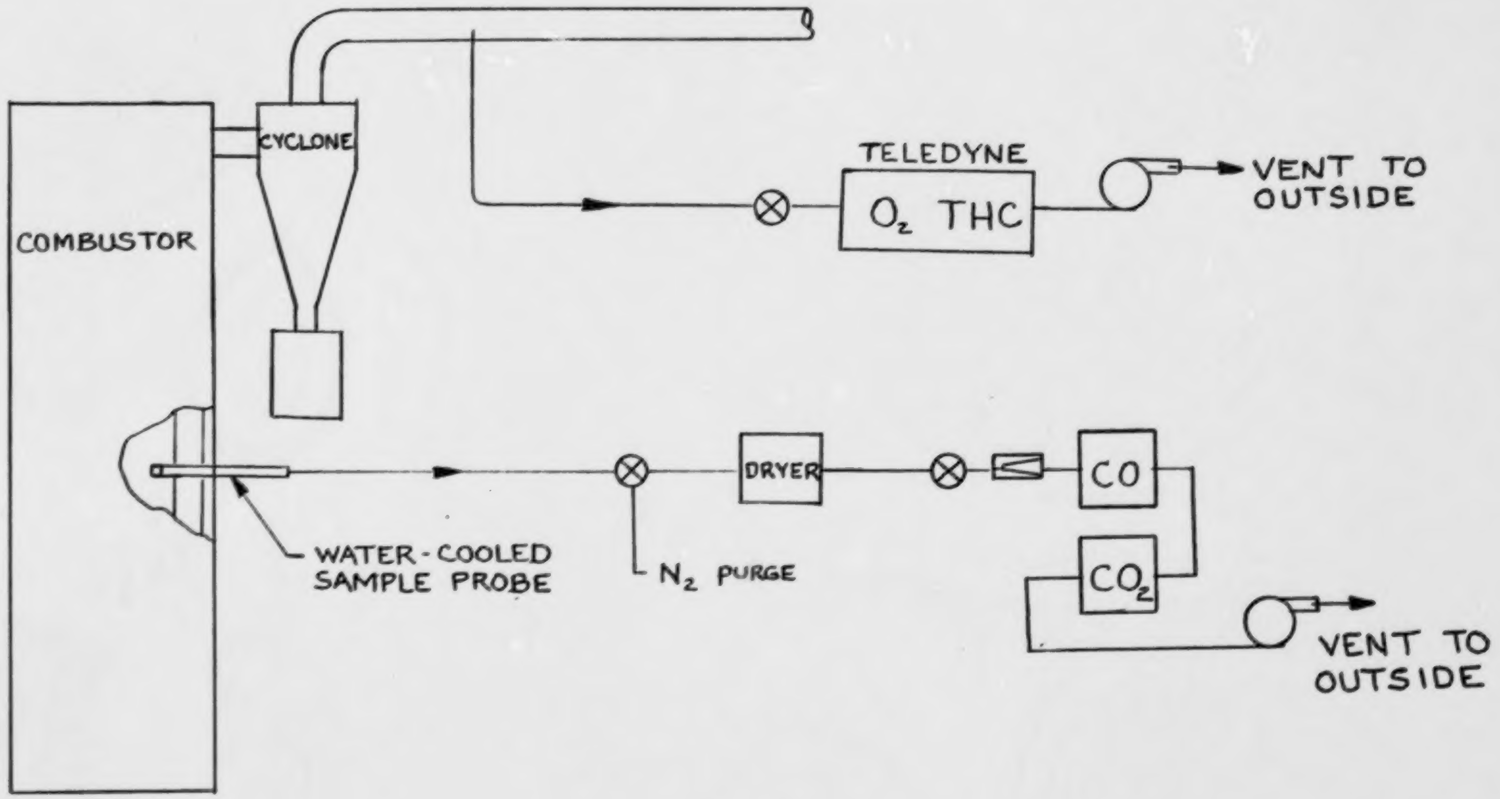

COMBUSTOR
CYCLONE
TELEDYNE
O_2 THC
VENT TO OUTSIDE
WATER-COOLED SAMPLE PROBE
N_2 PURGE
DRYER
CO
CO_2
VENT TO OUTSIDE

Figure 2-9. Flue Gas Sampling System.

2.2 TEST METHODS

2.2.1 Facility Operation Parameters

With schedule constraints limiting the facility shakedown time, operating parameters were arbitrarily chosen which would meet the goals of the program yet remain achievable with a moderate degree of confidence.

The original operating parameters were based on a bed expansion of 100 percent. Early shakedown runs indicated that an 18-inch slumped bed remained below 24" when fluidized. In order for the gas burner to impinge on the bed to aid in pre-heat and to attain a 36-inch fluidized bed height, bed material was added to a slumped height of 26 inches. Bed material was then seen bubbling up past and filling the viewport tube located 36 inches above the distributor plate.

The added bed material caused other parameters to increase significantly over planned values. The air flowrate and superficial velocity doubled requiring a like response in fuel feed rate. The bed temperature was reduced to 1800°F because the downstream sampling location temperature was higher than expected due to the higher mass flowrate of air.

The final operating parameters chosen for Phase I testing are summarized below:

Bed Material	Spherical bauxite beads 85-90% Alumina (Al_2O_3) dia. = .035 in. density = 135 lb/ft^3(bulk)
Bed Height	26 inches slumped, 3 feet expanded
Bed Temperature	1800-1850°F
Superficial Velocity	3.8 feet/second
Apparent Residence Time	0.8 seconds
Fuel Feed Rate	1.6 gallons/hour
Air Flowrate	60-70 standard cubic feet per minute
Excess Air	150-200%

2.2.2 Test Matrix

Six sampling runs were made with two different fuel waste mixtures burned. The test matrix is shown in Table 2-2.

TABLE 2-2. TEST MATRIX

Test	Run	Date	Test Fuel Mixture	Sampling Methods
1	1	5/05	#2 fuel oil w/ 2% Triphenylmethane	1. Source Assessment Sampling System (glass (SASS)
	2	5/10	1% 4-Nitrotoluene 1% Isophorone	
	3	5/12	1% α,α,α,p-Tetrachlorotoluene *	2. Stack Dilution Sampling System (SDSS)
2	4	5/17	#2 fuel oil w/ 2% Tetrahydrofuran	Volatile Organic Sample Train (VOST)
	5	5/17	2% Methyl Vinyl Ketone	
	6	5/17		

Sample volumes and times for each method are defined in Section 3, Sampling and Analysis Methods.

*One of the non-volatile compounds planned for the test, 2,4,6,Triphenoxy-s-Triazine, was not included because it failed to dissolve in the fuel oil during laboratory tests.

The decision was made to conduct the three VOST runs on the same day rather than three consecutive days as planned. This decision was based on the hardship on the facility during start-up and the short sampling time (~1 hr) required by the VOST. An added advantage was gained by the reduction in handling of the VOST glassware, minimizing the breakage.

2.2.3 Fuel/Waste Mixture Preparation

The test compounds were added to the fuel oil according to plan by pouring the chemicals into a 30-gallon drum containing the No. 2 fuel oil. This procedure was accomplished by two fully-protected personnel, one representative from EPA-TSS and one from Acurex, with a third man dressed outside the fuel shed to help if necessary. Each mixture was prepared on the morning of the first run with that test mixture. Fuel/waste samples were taken three times per run day at the beginning, middle and end of run. This activity is more completely discussed in the health and safety section of this report (Section 5).

2.2.4 Facility Operation

2.2.4.1 Pre-Test

All facility equipment was checked out and all system safety interlocks were verified. Due to some eleventh-hour problems such as a burst water pressure line in the control console, certain instrumentation, primarily the magnehelic gauges, were not calibrated prior to the test but were properly adjusted during the test period. The raw pressure data from magnehelics which were out of calibration at the start of the test were corrected in the operational data presented in Appendix E.

2.2.4.2 Start-Up

Modifications were made to the start-up procedures due to findings from the early shakedown runs. Standard start-up procedures evolved with each successive facility operation. The blower and electric air heaters (set @800°F) were turned on the evening before each test date. The gas burner was also operated overnight at a firing rate of 110,000 Btu/hr. On the following morning, temperatures downstream of the combustor would be near operating levels while the bed would be about 500°F. The electric heaters would then be adjusted up to their maximum settling of 1100-1150°F.

Plans called for heating the bed to 1000°F with the gas burner. The large air flow requirements for fluidization of the deeper bed made that procedure slow. Excessive blower power requirements for the cold bed (600°F) forced the injection of clean oil to speed the heat up of the unit. The oil did not ignite until it reached the gas flame but the bed temperature steadily increased. Once the bed temperature reached 1200°F, temperatures above the bed began to decrease indicating that the bed had reached the auto-ignition temperature of the oil. Bed temperatures rose to within 50°F of the temperature above the bed and remained as long as proper operation continued.

The gas burner may be turned off at any time after the bed reaches 1500°F. Standard procedure became to leave the gas burner on and overshoot the desired operating temperature by 30°F. When the gas was shut off the temperature dropped back to near the desired value.

2.2.4.3 Operation

Upon reaching the desired operating temperature of 1800°F, the air flowrate was adjusted to the proper setting to maintain fluidization. After conditions stabilized, the three-way fuel selector valve was switched to spiked oil. The fuel flow control valve was adjusted if necessary to maintain temperature in the bed. When steady state conditions (i.e. no fuel rate adjustments were required to maintain temperature) were achieved and announced by the operator, flue gas sampling began.

At the end of the test period, the three-way valve was switched back to clean fuel for 15 to 30 minutes in order to purge the fuel oil nozzle prior to shutdown.

No special shutdown procedures were found to be necessary. After the nozzle purge was completed, the three-way valve was turned to a closed position. The shutoff valve was kept in the open position until no pressure was indicated in the downstream fuel line. The fuel feed system was shutdown including the pumps and shut-off valve. When the oil feed stopped, an air purge valve opened to clear the oil out of the nozzle and to keep fine particles from plugging the nozzle holes. The electric heaters were shut-off and the bed was allowed to cool with only the blower operating.

2.2.4.4 Data Collection

All collected data were manually recorded at one hour intervals. System temperatures were automatically recorded with a 24-channel chart recorder but the recorder malfunctioned during Run 3. Continuous monitors were used to measure CO and CO_2 concentrations in the freeboard flue gas. These values were documented by a 2-channel chart recorder.

2.2.4.5 Post-test

Upon completion of the test series, solids samples were taken of the bed material, primary cyclone catch, and baghouse catch. These samples were analyzed to determine if any of the test compounds were present in detectable quantities. A size distribution was performed on the bed material after it was determined to be free of contaminants. Size comparisons were made with clean unused bed material to evaluate the erosion effects of the fluidized bed. Solids samples were not taken until the end of the test program to minimize personnel exposure if hazardous compounds were present.

The drum which held the test fuel was cleaned by twice rinsing with acetone and vacuuming the contaminated solvent into "polybottles" for disposal in EPA's on-site hazardous waste incinerator. The drum was then filled with clean fuel oil and the facility operated for three hours to flush out the waste fuel system.

The plenum was dropped from the combustor section to examine the condition of the distributor plate and to measure the loss of bed material through the distributor into the plenum which occurred each time the facility was shutdown.

Operational data were evaluated to determine any characteristics of the facility which suggest improvement for future phases.

SECTION 3

SAMPLING AND ANALYSIS METHODS

3.0 INTRODUCTION

This portion of the report summarizes tests for volatile, semivolatile, and condensable emissions conducted by EPA/TSS and Southern Research Institute on the atmospheric fluidized bed incineration unit.

The sampling and analytical effort for Phase I of the Fluidized Bed Incineration Program of AEERL was developed to accomplish three major goals:

1. Provide experience to AEERL project officers in the advantages and disadvantages of sampling and analytical methods proposed for hazardous waste incineration.
2. Provide an opportunity to set up and "debug" sampling and analytical efforts planned for Phase II and Phase III of the effort.
3. Provide an indication of the destruction efficiency of the on-site fluidized bed incinerator using a group of surrogate principal organic hazardous constituents.

3.1 SAMPLING METHODS DESCRIPTION

Three discrete approaches to flue gas measurement were used. Each method addressed a different facet of hazardous waste incineration flue gas sampling. The three areas of interest are volatile, semivolatile and condensable organic sampling. Continuous monitoring for CO, CO_2, etc. is not addressed in this part of the report.

3.1.1 VOST

The Volatile Organics Sampling Train (VOST), shown in Figure 3-1, was used to sample volatile organic materials from the combustion flue gas stream. Tetrahydrofuran and methyl vinyl ketone were the volatile surrogate wastes sampled. These two volatile compounds have boiling points between 30° and 100°C (Table 3-1) which allows testing of the Volatile Organic Sampling Train (VOST).

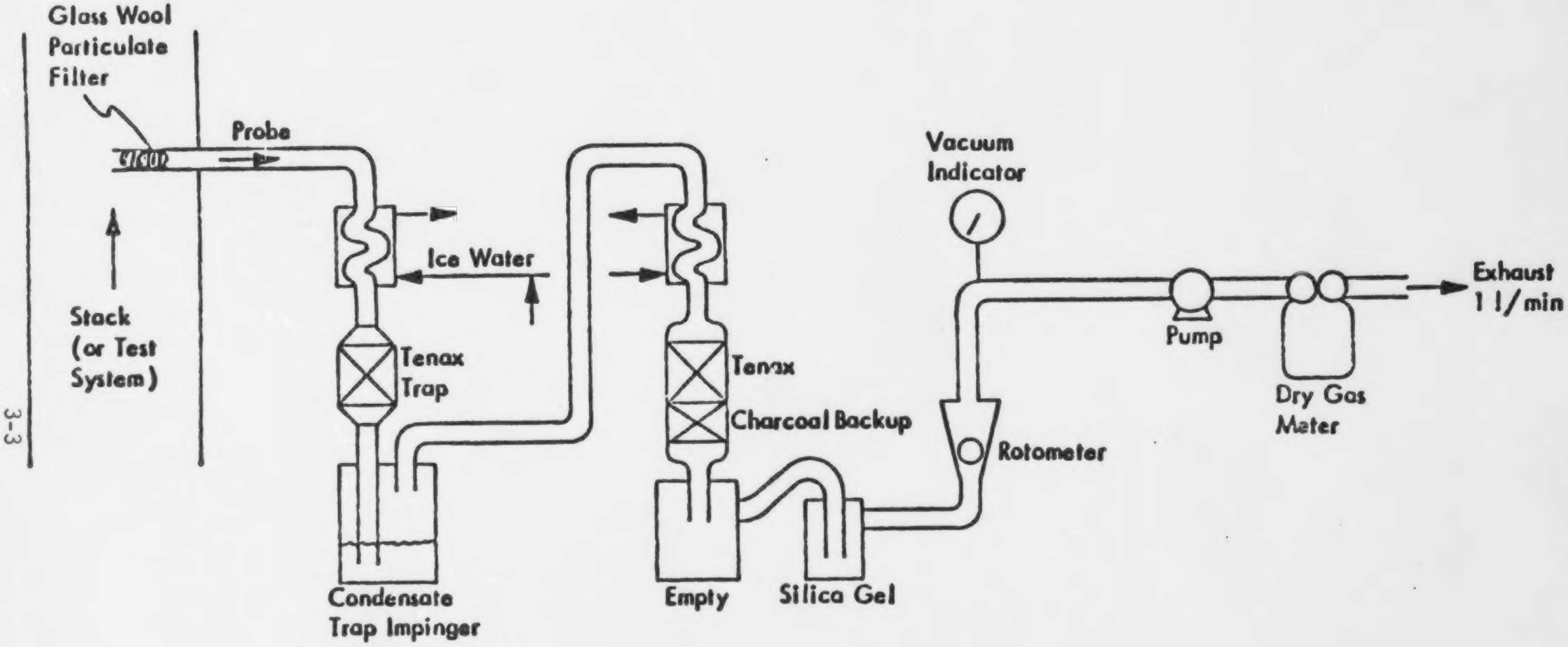
TABLE 3-1. BOILING POINTS AND HEAT OF COMBUSTION FOR VOLATILE SURROGATE WASTE

Volatile Compound	B.P.	* Hc/g
Tetrahydrofuran	64-65°C	8.43 kcal/g
Methyl Vinyl Ketone	79-80°C	7.19 kcal/g

*Heats of combustion (Hc) Kcal per gram of compound computed by method of Handrick, Ind. Eng. Chem. 48:1366 (1956).

Choice of volatile compounds was restricted to materials not presently on the potential principle organic hazardous waste list (RCRA Appendix 8) and to compounds which did not contain halogen substituents. As a consequence of these restrictions both the compounds chosen were water soluble. Tetrahydrofuran is a common laboratory solvent with minimal health hazard (Table 3-2). Methylvinylketone is a lachrymator, with its associated handling problems (Table 3-3).

Samples were acquired using a VOST assembled by Envirodyne Engineers based on the design concept of L. Johnson and J. Harris (Fig. 3.1). A detailed description of the train construction and resin preparation is given in Appendix F. The train was configured in three sections. The probe section consisted of a pyrex probe connected via balljoint to a threaded teflon vacuum valve. The organic sorbent section consisted of two serial sorbent modules each preceeded by a temperature controlled cooling coil. The first organic module contained only Tenax GC^{R*} and was followed by a condensate trap. The second organic module contained


Glass Wool
Particulate
Filter
Probe
Stack
(or Test
System)
Ice Water
Tenax
Trap
Condensate
Trap Impinger
Tenax
Charcoal Backup
Empty
Silica Gel
Vacuum
Indicator
Rotometer
Pump
Dry Gas
Meter
Exhaust
1 l/min

Note: Tenax & tenax/charcoal traps were 1.6 cm in diameter & 10 cm long

Figure 3-1. Volatile Organic Sampling Train (VOST).

TABLE 3-2. TETRAHYDROFURAN PROPERTIES

- Description
  - Colorless liquid with ether-like odor
  - Highly flammable
  - bp: 64-65°C
  - fp: -17°C
- Threshold Limit Values TLVs
  - TLV-TWA: 200 ppm 590 mg/m^3
  - TLV-STEL: 250 ppm 735 mg/m^3
- Toxic Effects
  - Irritates eyes and respiratory system
  - Has narcotic effect
  - May damage liver and kidney

TABLE 3-3. METHYL VINYL KETONE PROPERTIES
$CH_3\text{-}C\text{-}CH\text{-}CH_2$

- Description
  - Colorless liquid with irritating odor
  - Dangerous near heat, flame or oxidizers
  - bp: 79-80°C
  - fp: -6°C
- Toxic Effects
  - Irritant to skin, eyes, mucous membrane
  - Lachrymator

Tenax GC^{R*} and petroleum based charcoal. The final section of the train consisted of the various traps, meters and gauges required to dry the gas stream and measure the volume of gas sampled. The VOST was operated at approximately 1 liter/minute for 60 minutes during which time the Tenax -GC^{R} and Tenax - GC^{R}/Charcoal cartridge pair was changed at 20 minute intervals. End caps were sealed in clean aluminum foil while cartridges were being installed in the VOST.

The actual sampling with the VOST was modified from that proposed in the work plan. The plan called for three separate sampling runs over a period of three days. To shorten the period of the tests and allow sampling of the FBC/I test rig with minimum possible process variation, three 60 minute VOST runs were scheduled for one day. Samples from a run consisted of three pairs of VOST cartridges each containing the volatile organic material and a separate container of condensate from 20 liters of process gas. The three sets of three VOST cartridge pairs and associated condensate were acquired sequentially over one day's sampling. No attempt was made to rinse the sampling probe or condenser units between the 60 minute (3 pair + condensate) runs.

The final VOST run was shortened to two pairs of cartridges (40 min) and the one remaining pair of cartridges was used as a combined blank for the day's sampling. No other changes were made in the VOST operation.

3.1.2 SASS

A Source Assessment Sampling System (SASS) (Figure 3-2 and 3-3) equipped with a corrosion-proof glass sorbent module and all glass probe, filter, and cyclone assembly was used to sample organic compounds with boiling points greater than 100°C. Three semivolatile compounds (Table 3-4) were chosen to

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*Tenax-GC is a registered trade mark by ENka Research Institute Arnhem.

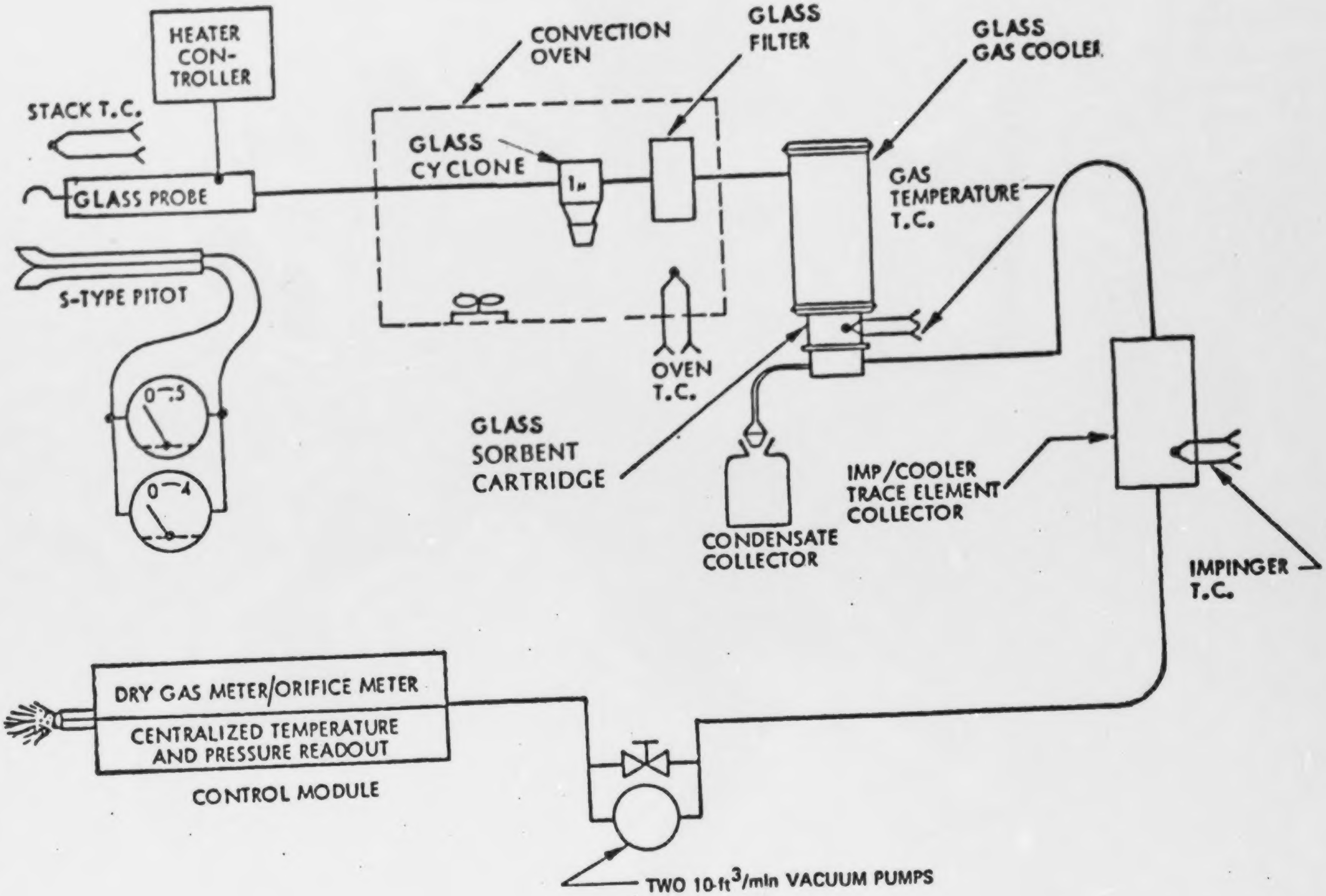
HEATER CON-TROLLER
STACK T.C.
GLASS PROBE
S-TYPE PITOT
0–.5
0–4
CONVECTION OVEN
GLASS CYCLONE
1μ
GLASS FILTER
OVEN T.C.
GLASS SORBENT CARTRIDGE
GLASS GAS COOLER
GAS TEMPERATURE T.C.
CONDENSATE COLLECTOR
IMP/COOLER TRACE ELEMENT COLLECTOR
IMPINGER T.C.
DRY GAS METER/ORIFICE METER
CENTRALIZED TEMPERATURE AND PRESSURE READOUT
CONTROL MODULE
TWO 10-ft³/min VACUUM PUMPS

Figure 3-2. Glass Source Assessment Sampling System (Glass SASS)

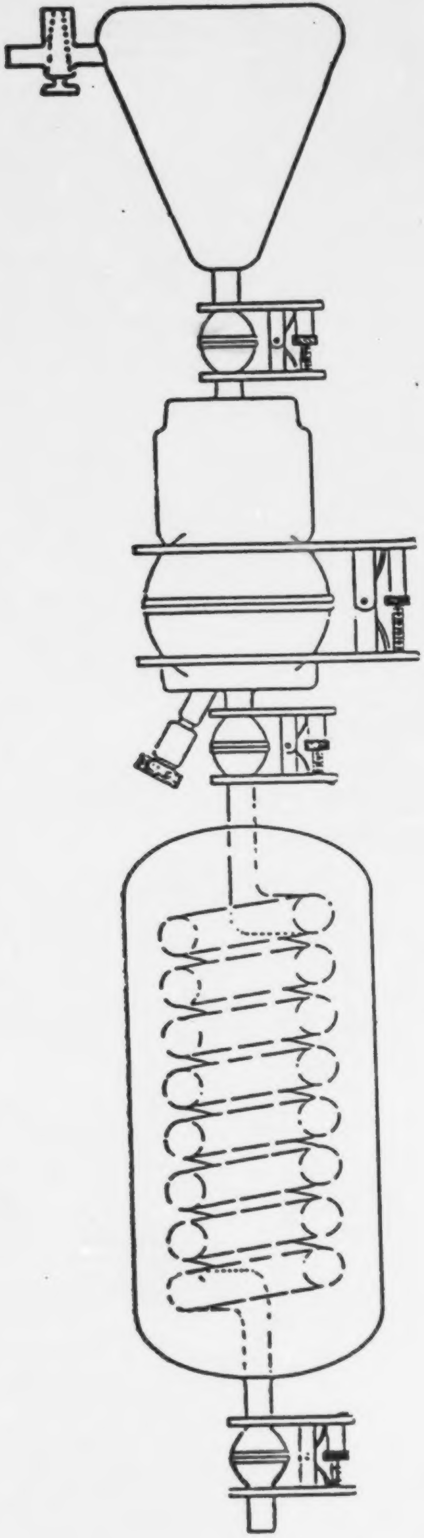

Figure 3-3. Glass Module Assembly for SASS Train

test the sorbent sampling train (Glass SASS): Isophorone, 4-nitrotoluene, and α,α,α,p-tetrachlorotoluene. In addition, the condensable compound triphenylmethane was sampled, so that the results from the SASS sampling could be compared to the results from the dilution sampling described below.

TABLE 3-4. BOILING POINTS AND HEAT OF COMBUSTION FOR SEMIVOLATILE AND CONDENSIBLE SURROGATE WASTE

Semi-Volatile	B.P.	*Hc/g
α,α,α,p-Tetrachlorotoluene	245°C	3.12 kcal/g
4-nitrotoluene	238°C	6.48 "
Isophorone	213°C	8.75 "
Non-Volatile		
Triphenylmethane	358.9	9.72 "

Choice of these compounds was restricted to materials not presently on the potential principle hazardous waste list (RCRA Appendix 8) just as was the case for the choice of volatile compounds. One chlorinated compound was chosen for this group, α,α,α,p-Tetrachlorotoluene. Description of the compounds and their health effects can be found in Tables 3-5, 3-6, 3-7, and 3-8.

SASS operation followed standard procedures with the exception of the particle collection hardware. A glass filter holder and glass cyclone were substituted for the heated steel cyclone and filter holder used in the standard SASS. The glass cyclone and filter assembly used were part of a standard Method 5 sampling train. The filter was heated by a mantle to minimize water condensation.

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*Heats of combustion (Hc) Kcal per gram of compound were computed by the method of Handrick, Ind. Eng. Chem. 48:1366 (1956).

TABLE 3-5. 4-NITROTOLUENE PROPERTIES

- Description
  - Yellow solid with aromatic odor
  - Insoluble in water
  - mp : 53°C
  - fp : 106°C
  - bp : 238°C
- Threshold Limit Values TLV

  TLV-TWA: 2ppm 1 mg/m^3
- Toxic Effects
  - difficulty breathing
  - cyanosis
  - unconciousness in severe cases
  - injures eyes if taken by mouth

TABLE 3-6. ISOPHORONE PROPERTIES

- Description
    - Colorless liquid with camphor-like odor
    - Slightly soluble in water
    - mp : -80°C
    - bp : 213-214°C
    - fp : 84°C
- Threshold Limit Values TLVs
    - TLV-TWA : 5 ppm 25 mg/m^3
- Toxic Effects
    - Irritates eyes and respiratory system
    - May cause corneal damage
    - Irritates the skin
    - Toxic if ingested

TABLE 3-7. α,α,α,p-CHLOROTOLUENE PROPERTIES

- Description
  - Colorless or yellow liquid
  - Insoluble or slightly soluble in water
  - bp : 245°C
  - mp : −8°C
- Threshold Limit Values (TLVs)
  - TLV-TWA : 1-50 ppm (range of class)
  - TLV-STEL : up to 75 ppm (range of class)
- Toxic Effects
  - Irritates eyes, skin, respiratory system

TABLE 3-8. TRIPHENYL METHANE PROPERTIES

- Description
  - mp : 93°C
  - bp : 359°C
  - fp : 100°C
- Threshold Limit/Value (TLV)
  - unavailable
- Toxic Effects
  - unavailable

Three sampling runs were performed on three separate days. Each run accumulated approximately 30 dry standard cubic meters (dscm) of stack effluent. Four samples were recovered from each SASS run:

1. Probe, filter, cyclone and probe rinse catch.
2. XAD-2 resin and module rinse
3. Condensate from XAD-2 module
4. Impinger solutions (as one combined sample)

3.1.3 SDSS

The Stack Dilution Sampling System (SDSS), Figure 3-4, was used to sample condensable organic material. Triphenyl methane and 2,4,6,triphenyl triazine were chosen for the FBC incineration tests but the triphenyl triazine was found to be insoluble in fuel oil. Therefore, triphenyl methane remained as the only organic material spiked into the fuel which would be sampled and retained by the SDSS.

The condensible testing performed with the SDSS followed procedures developed for inhalable particulate (IP) testing. Both the device and protocol are described in Appendix G. Briefly, sample is drawn through the system at approximately 23 liters/minute (0.8 ft^3/min) for 4 to 5 hours. Cool dry dilution air at approximately 25:1 mixes with the sample stream. After a residence time of approximately 6 sec. the particulate material is collected on a teflon membrane filter.

After sampling, the cyclones were unloaded and cyclone particulate catches collected. The probe, heated hose, and sample gas inlet assembly of the SDSS were washed with solvent (dichloromethane) and retained for chemical analysis. The filters are desiccated, weighed and retained for preparation and analysis.

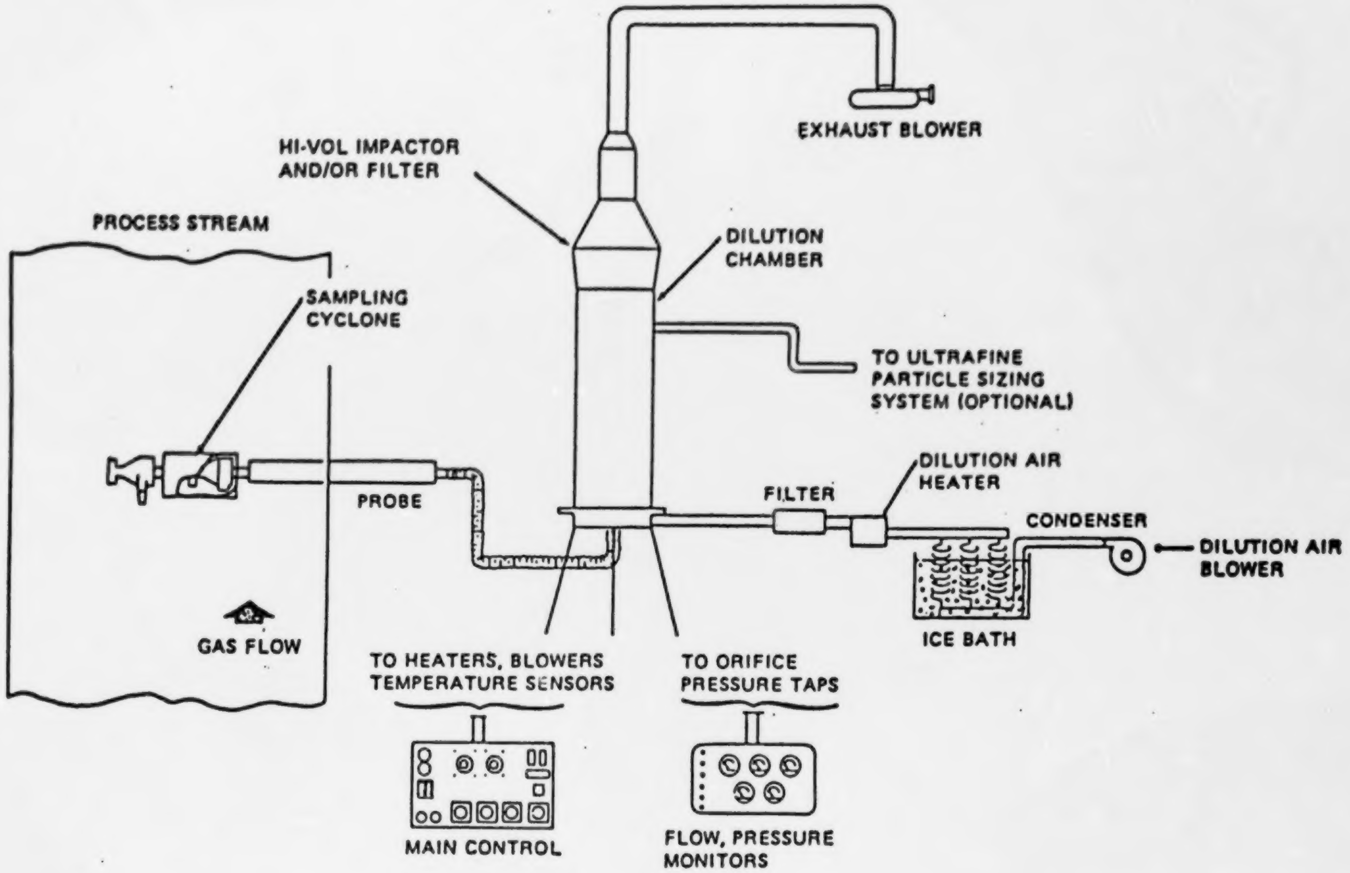
EXHAUST BLOWER
HI-VOL IMPACTOR AND/OR FILTER
PROCESS STREAM
DILUTION CHAMBER
SAMPLING CYCLONE
TO ULTRAFINE PARTICLE SIZING SYSTEM (OPTIONAL)
DILUTION AIR HEATER
FILTER
PROBE
CONDENSER
DILUTION AIR BLOWER
ICE BATH
GAS FLOW
TO HEATERS, BLOWERS TEMPERATURE SENSORS
TO ORIFICE PRESSURE TAPS
MAIN CONTROL
FLOW, PRESSURE MONITORS

Figure 3-4. Stack Dilution Sampling System

3.2 SAMPLE PREPARATION

The analyses procedures described here include all sample handling and compound recovery techniques applied to samples after they had been acquired from the FBC process stream. In some cases sample handling was as simple as storage (e.g. VOST samples). In other cases, sample preparation required steps including extraction and concentration prior to the actual analytical finish. Preparation is discussed in three section by sample type: fuel samples, VOST samples, and SASS/Dilution tunnel samples.

3.2.1 Volatile Spiked Fuel Handling

Volatile compounds THF and MVK were spiked into one batch of No. 2 fuel oil immediately preceeding the sampling run. Fuel samples representative of fuel feed during VOST runs were collected in 100 ml screw cap glass bottles cleaned according to reference 1.* Feed was tapped at the beginning, middle, and end of the sampling run. Three samples resulted, each sample filled the sample bottle completely with minimum head space remaining. During sample acquisition, unique identifiers were not placed on sample bottles, resulting in unknown timing of the three discrete samples. Therefore, actual field samples from the combustion stream can only be compared to the average value of spiked material in the fuel samples.

3.2.2 Semivolatile and Condensible Fuel Storage

Semivolatile and condensible surrogate waste compounds were spiked into one batch of No. 2 fuel oil. Three Erlenmeyer flasks with ground glass stoppers were cleaned according to the procedures given in reference 1 and used for spiked fuel sampling. Three 100 ml aliquots of fuel were taken during each sampling run. One aliquot was taken at the beginning of sampling, one at the middle and one at the end of each sampling run. The total composite sample for each run was labeled and transported to the laboratory in a cooler partially filled with sawdust which is described in the program safety plan. Samples were stored at ambient temperature in a dark ventilated area under a hood in the analysis laboratory. A total of three 300 ml samples were acquired, each sample representing a composite of fuel from the time period used to take each stack sample.

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*Reference 1. Lentz, D. E. et al., IERL-RTP Procedures Manual: Level 1 Environmental Assessment (2nd Edition). EPA 600/7-78-201.

3.2.3 VOST Cartridge

After sampling, VOST cartridges were resealed with their original caps and wrapped in aluminum foil. Cartridges were transported to the analytical laboratory and stored in a refrigerator-freezer at 0°C.

3.2.4 SASS and Dilution Tunnel Sample Storage

After sampling, train component openings were sealed temporarily with aluminum foil and transported to the analytical laboratory. Samples were recovered from probe, filter housings, and XAD-2 module by rinsing train components into appropriate storage bottles. All liquid samples were stored at 4°C in the dark. SASS and Dilution Tunnel filters were removed from their holders and desiccated.

After weighing, SASS filter samples were stored in the desiccator in the dark until extraction for analysis. Dilution Tunnel samples were stored in glass sample bottles in the dark at 4°C before preparation for analysis. Extraction was performed within one week of receipt.

3.2.6 Sample Preparation and Extraction of Semivolatile and Condensible Organics

3.2.6.1 Semivolatile Sample Preparation

Samples from the glass SASS were combined and spiked with D_{10}-anthracene before extraction at known concentrations of approximately 200 μg/ml per reference 1.

Probe rinse solids, cyclone rinse solids, filter housing rinse solids and filter samples were evaporated to dryness, weighed, combined, spiked with d_{10}-anthracene and extracted with dichloromethane in a Soxhlet extractor for

24 hours. This combined sample was concentrated to exactly 1.0 ml, placed in a clean septum sealed serum vial and stored at 4°C in the dark until analysis. Analyses were performed 1 to 3 months after extraction.

XAD-2 resin and resin module rinse were combined, spiked with d_{10}-anthracene and extracted with dichloromethane in a Soxhlet extractor for 24 hours. The combined sample was concentrated to exactly 1.0 ml, placed in a clean septum sealed serum vial and stored at 4°C in the dark until analysis. Analyses were performed 1 to 3 months after extraction.

The volume of the resin module condensate (~1200-1400 ml) was measured and the condensate was divided into two samples, one of exactly 10 ml set aside for chloride (Cl^-) analysis by ion chromatography and the remainder for extraction and organic analysis. Extraction was accomplished by solvent partition at pH 2 and pH 12. The organic portion from these condensate extractions was combined and concentrated to exactly 1.0 ml per Reference 1. Sample storage involved placing the concentrated extract in a septum sealed serum vial and storage in the dark at 4°C.

Impinger 1 was used empty, impinger 2 contained deionized water (500 ml), and impinger 3 contained 0.1 N NaOH (500 ml). Impinger solution volumes were measured, samples were placed in glass sample bottles and stored in the dark at 4°C until Cl^- analysis could be performed. Chloride analyses were performed within 2 weeks of sample receipt. Organic analyses were performed 3-4 months after receipt.

3.2.6.2 Dilution Tunnel Samples

The probe, heated hose, and sample gas inlet assembly of the SDSS were washed with dichloromethane to recover condensed sample. Filters were desiccated in a polythylene glove bag (I^2R Models SS-1 and x-37-37 placed in series). The first bag served as an introductory chamber, while the second bag housed the samples and the balance. An air lock between the two bags allowed for deposition of samples into the introductory chamber without contaminating the dry air of the larger bag. The samples were then weighed and stored in a dry atmosphere, thereby reducing the hygroscopic weight gain as much as possible.

In order to provide a chemically clean substrate for chemical analysis and to inhibit gas-filter reactions, teflon membrane filters were used on the SDSS. GHIA Zefluor filters with 2 μm pore size have proven satisfactory in previous tests, and operate with no noticeable problems in the SDSS. After the filters were weighed, they were transferred to glass wide mouth bottles and transported to the analysis laboratory.

Extraction of dilution tunnel samples included spiking with a known quantity of d_{10}-anthracene and Soxhlet extraction for 24 hours with methylene chloride. One sample from each dilution tunnel run was prepared from the composite of the probe wash and filter catch. The extract was concentrated to 10 mililiters using the Kaderna Danish technique. Concentrating from 10 mililiters to exactly one mililiter was done under a stream of purified nitrogen transferred to a septum sealed glass vial and stored at 4°C until analysis could be performed. Analyses were performed 3 to 4 months from sample receipt.

3.3 VOLATILES ANALYSIS

Analysis of VOST samples was accomplished using the modified method for purgeable organic compounds, EPA Method 624, described below. Volatile compounds were desorbed from porous polymer resin and charcoal into a purge and trap apparatus. The analytical finish was performed by GC/MS. Full details of the report and a general protocol for VOST sampling and analysis are provided in Appendix F.

3.3.1 Description of the Analytical Apparatus

The analtyical apparatus consisted of three major components: a Nutech 320 thermal desorption unit, a Hewlett Packard 7675A purge and trap unit, and a Hewlett Packard 5993 GC/MS/DS. A schematic of the apparatus configuration is shown in Figure 3-5.

The "as received" Nutech Model 320 was modified by rerouting the gas flow to bypass the cryogenic trap and to allow the sample cartridge holder to be vented after desorption. Absorption gas supply was provided from the Hewlett Packard 7675A during the purge mode and materials desorbed from the sampling cartridges were fed into the purge supply tube of the 7675A purge and trap unit.

3.3.2 Description of Method Evaluation Efforts

Initial tests with standards using the thermal desorption unit and federal register EPA Method 624 showed that the surrogates chosen for this set of tests were not amenable to analyses without modification. Studies were conducted with each part of the analytical apparatus to determine where the compounds were being lost. The compounds were detected by direct injection into the GC/MS column and by injection in methanol solution in a dry purge and trap tube. The compounds also were detected using a spiked tenax trap as the compound source and a dry purge and trap tube. However, tetrahydrofuran and methylvinyl ketone spiked onto the Tenax cartridges could not be purged and detected when water was present in the purge tube. Three analysis parameters are variables in method 624: purge tube temperature, purge time, and the quantity of water in the purge tube. By extending the purge time and reducing the amount of water initially placed in the purge tube, consistant results for the internal spike (methyl furan) and the two surrogate wastes (tetrahydrofuran

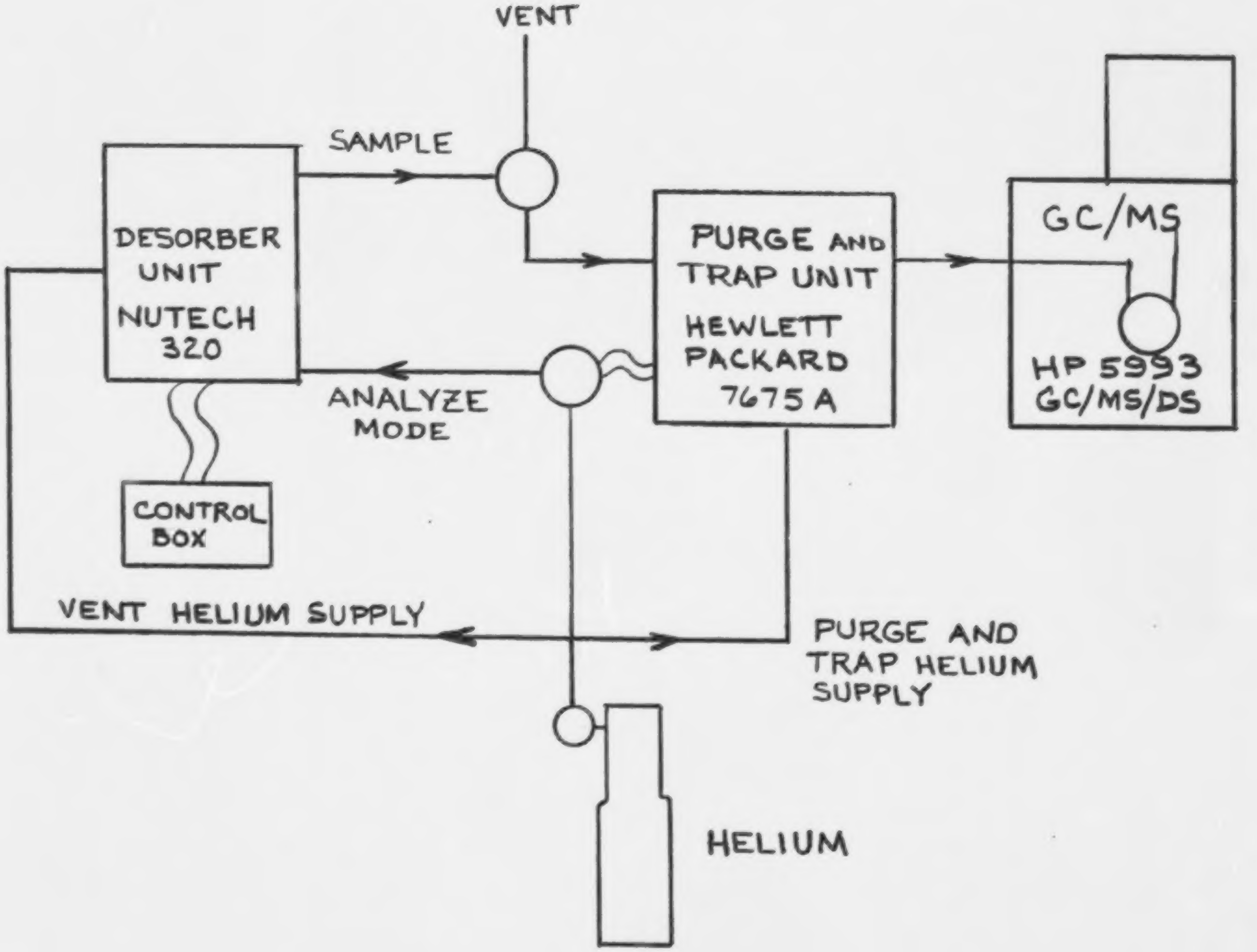
VENT
SAMPLE
DESORBER UNIT
NUTECH 320
ANALYZE MODE
CONTROL BOX
PURGE AND TRAP UNIT
HEWLETT PACKARD 7675 A
GC/MS
HP 5993 GC/MS/DS
VENT HELIUM SUPPLY
PURGE AND TRAP HELIUM SUPPLY
HELIUM

Figure 3-5. VOST Analytical Equipment.

and methyl vinyl ketone) were obtained. Further studies were performed to determine the purge time required to completely deplete the purge tube of volatile compounds. These studies resulted in the analytical conditions listed in Table 3-9.

3.3.3 Method Description

The sealed VOST cartridges from test runs were stored in the freezer compartment of a clean sample refrigerator. One-half hour prior to analysis, the cartridges were removed and allowed to thaw and warm to room temperature (~25°C). The inner cartridge containing the Tenax was then removed with teflon-coated tweezers, spiked with 2.0 microliters (102 nanograms) of 2-methyl furan, and immediately placed in the desorber unit. With minimum delay the purge-and-trap cycle was initiated and GC/MS control program was started. After the initial delay for purging, mass spectral data acquisition was begun and allowed to continue for approximately 30 minutes.

After data acquisition, peaks were identified using retention times and the eight most abundant ion masses characteristic of each compound. Base line correction was achieved by identifying masses characteristic of the compound of interest which were not subject to interferences, and correcting other indicative masses so that the ratio of ion intensities was correct for the compound of interest. That is, when the variation of the intensity of one of the masses being used was such that it was clearly at variance with the intensities of the other major masses in the spectra, it was corrected using the standard intensities for that compound. Baseline correction is particularly important for the analysis of low molecular weight compounds since background ions from the fragmentation of other materials in these source samples contribute to the ion abundance in the low molecular weight range.

Table 3-9. Analytical Conditions for VOST Cartridge Analysis

Desorbtion Parameters

Purge Time 50 minutes

Desorb time 6 minutes

Vent time 7 minutes

Auxillary line temperature 150°C

Desorb Temperature 180°C

Flowrate 5 ml/min.

GC/MS Parameters

Mass Range Scanned 30 to 100 AMU

Initial Oven temperature 32°C

Initial time 60 minutes

Program rate 8°C/minute

Final temperature 170°C

Total run time 85-90 minutes

EM Voltage 2400 EV

Time for total mass scan 0.9 sec

GC Column 2% Carbowax 1500 on
Carbopack B 60/80 mesh

Analysis of the Tenax-charcoal cartridges used as back-up was performed in the same manner as the Tenax cartridges. However, high background levels were encountered in both the control and sample cartridges. An examination of the trailing edges of these background peaks indicated the presence of both methyl vinyl ketone and tetrahydrofuran at the detection level of the instrument. Since the concentrations were below the quantitation limits, no quantitative results are provided for these samples.

3.4 SEMIVOLATILE AND CONDENSIBLE ANALYSIS

3.4.1 Glass SASS and Dilution Tunnel Analysis Methods Description

All extracts were concentrated to 1 ml and placed in septum-sealed vials for analysis. Each vial was spiked with 302 ng of dibromobenzene as an internal standard to verify instrument response. Samples were run in the following manner: 2 microliters of sample were injected into the GC/MS capillary column. The temperature program and GC/MS operating conditions are given in Table 3-10.

TABLE 3-10. GC/MS CONDITIONS FOR GLASS SASS AND SDSS SAMPLE ANALYSIS

Mass Range Scanned	40-400 amu
Initial Oven Temperature	50 deg C
Initial Time	5 minutes
Programming Rate	10 deg per minute
Final Temperature	250 deg C
Total Analysis Time	30 min
Fused Silica Capillary Column	SP 54
Split Injection - Split Ratio	10/1

The resulting chromatograms were acquired and a selected ion was plotted for masses indicative of triphenylmethane, isophorone, chlorobenzo-trichloride, nitrotoluene, bromobenzene and D_{10}-anthracene. When the indicator mass for these compounds was present, two additional masses characteristic of each compound were displayed and the relative abundance of the ions was compared to the mass spectra of the pure compounds injected as standards. All three masses were needed to confirm each compound. If the presence of the compound was confirmed, a response correction was performed based on the dibromobenzene internal standard, and the concentration of the compound was calculated.

SECTION 4

TEST RESULTS

4.1 OPERATIONAL DATA

Test runs were conducted on four days within a three week period. Appendix E shows the manually recorded operational data for the test series. The average pertinent values have been shown in Table 4-1 as a comparison of combustion conditions for each run. All of the test conditions were averaged from data recorded only during the sampling period. The data indicate that the combustor operating conditions were maintained within acceptable limits. The variance of temperatures in the bed between runs was within 20°C. The most noticeable variances were the reduction in fuel requirements from the beginning to end of each run and the reduction in pressure drop across the bed with each successive run. The former condition indicates that the facility had not reached steady state temperatures; also verified by increasing temperatures downstream. Figures 4-1 and 4-2 show system temperatures for Test 1 and 2 respectively, while Figure 4-3 indicates the range of temperatures from start to finish of a typical test day (Test 1, Run 2).

Combustion efficiency of the facility was not a concern of this program since incineration facilities are typically concerned with waste destruction rather than energy conversion. However, complete combustion of the fuel is essentially insured because of the excellent mixing properties and thermal homogeneity of fluidized beds and the large excess air required for their operation. Temperatures were maintained > 500°F above auto-ignition temperature of the fuel. Any computation of combustion characteristics of the facility would be difficult to obtain with precision due to the degree of accuracy of the raw data. The air and fuel flowrates were measured using flow obstruction devices (i.e. orifices, rotometers) with manually determined and recorded data. The high percentage of excess air required for fluidization made the concentrations of O_2, CO_2, CO and

Table 4-1. Facility Operational Data

Run	Plenum Temp	Combustion Zone Temp (in. above distributor)						Dist ΔP	Bed Density ΔP	Inlet Press	ΔP Inlet Orifice	Inlet Temp.	Air Flowrate (scfm)	Oil Flowrate (gph)	O_2 Concentration %
		5	12.5	22.5	32.5	37.5	42.5								
TEST 1															
1	684	1797	1785	1794	1810	1817	1773	27.8	37.1	70.6	5.83	411	71	1.52	12.9
2	628	1812	1802	1804	1816	1799	1725	27.8	34.8	66.9	5.16	400	66	1.63	12.6
3	648	1812	1804	1806	1825	1813	1744	28.0	34.2	65.0	5.11	405	65	1.48	12.7
TEST 2															
1	607	1831	1817	1819	1821	1796	1717	34.1	23.7	57.6	4.08	414	57	1.38	12.3
2	602	1832	1818	1823	1825	1800	1725	33.5	24.4	57.8	3.84	407	56	1.19	12.9
3	600	1833	1818	1825	1827	1803	1731	33.3	24.5	57.5	3.78	406	55	1.10	13.2

All temperatures in °F. Pressures in inches of water.

NO. 341-M DIETZGEN GRAPH PAPER
MILLIMETER

DIETZGEN CORPORATION
MADE IN U.S.A.

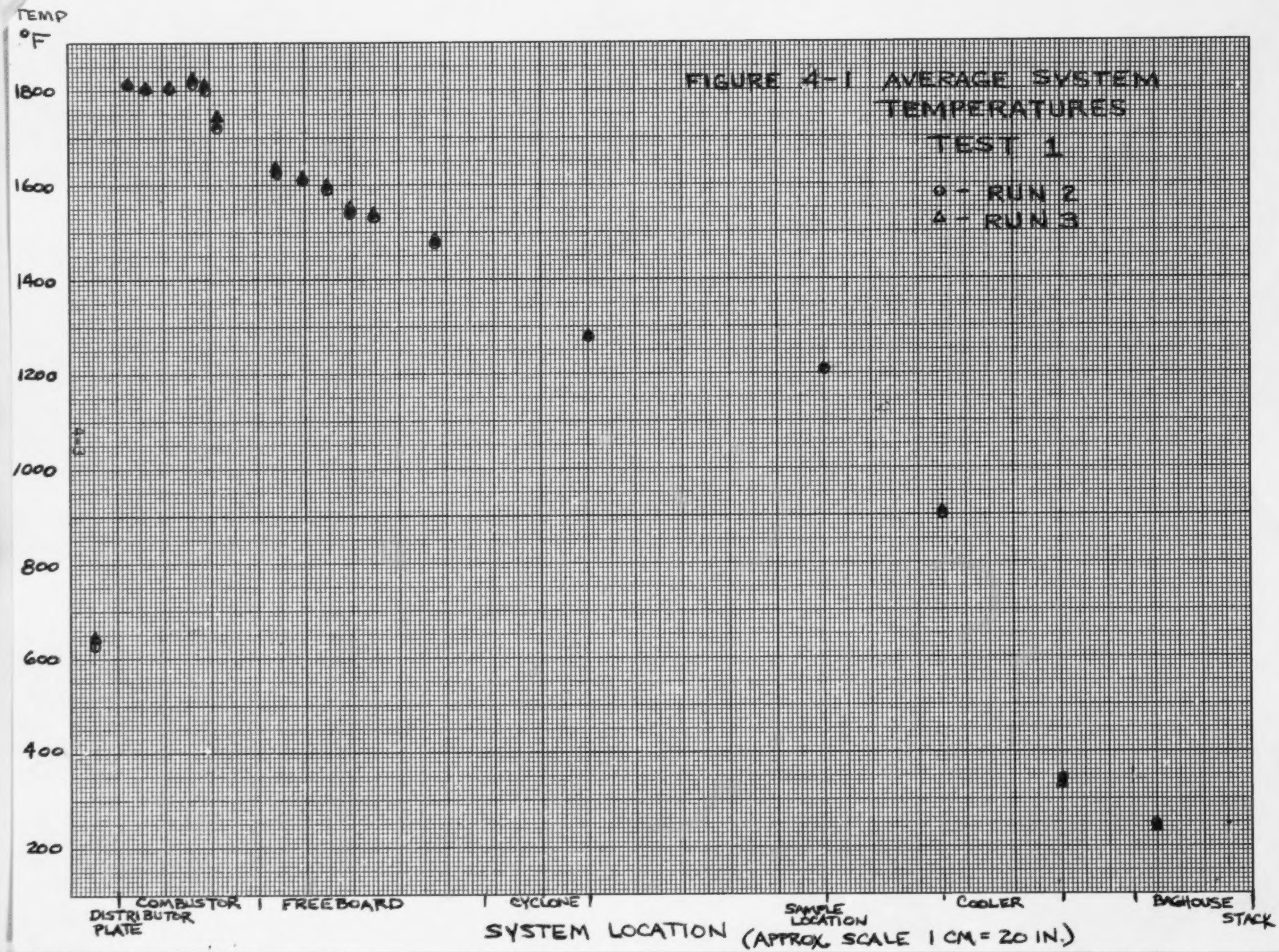

FIGURE 4-1 AVERAGE SYSTEM TEMPERATURES

TEST 1

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MILLIMETER

DIETZGEN CORPORATION
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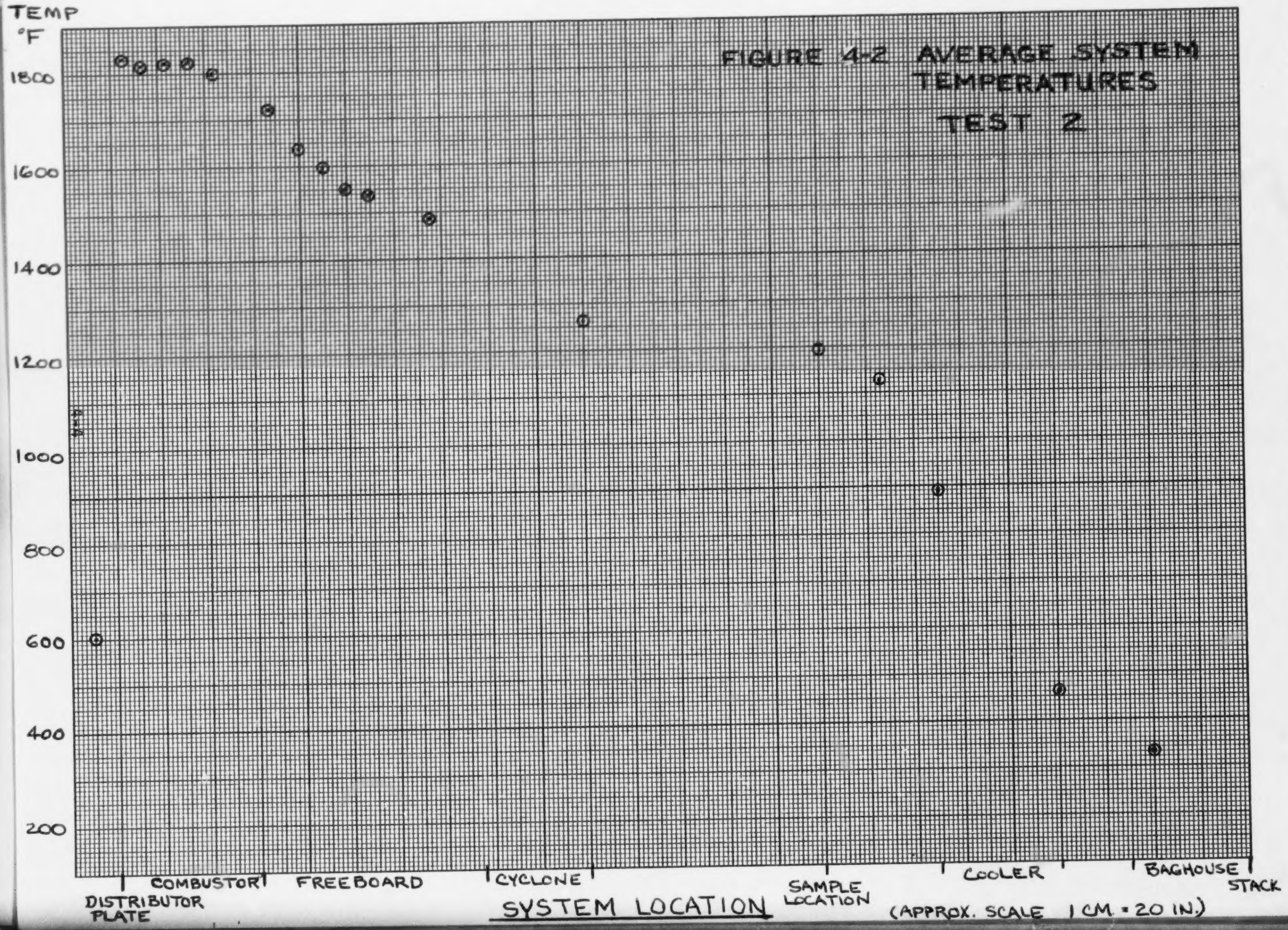


FIGURE 4-2 AVERAGE SYSTEM TEMPERATURES

TEST 2

NO. 341-M DIETZGEN GRAPH PAPER
MILLIMETER

DIETZGEN CORPORATION
MADE IN U.S.A.

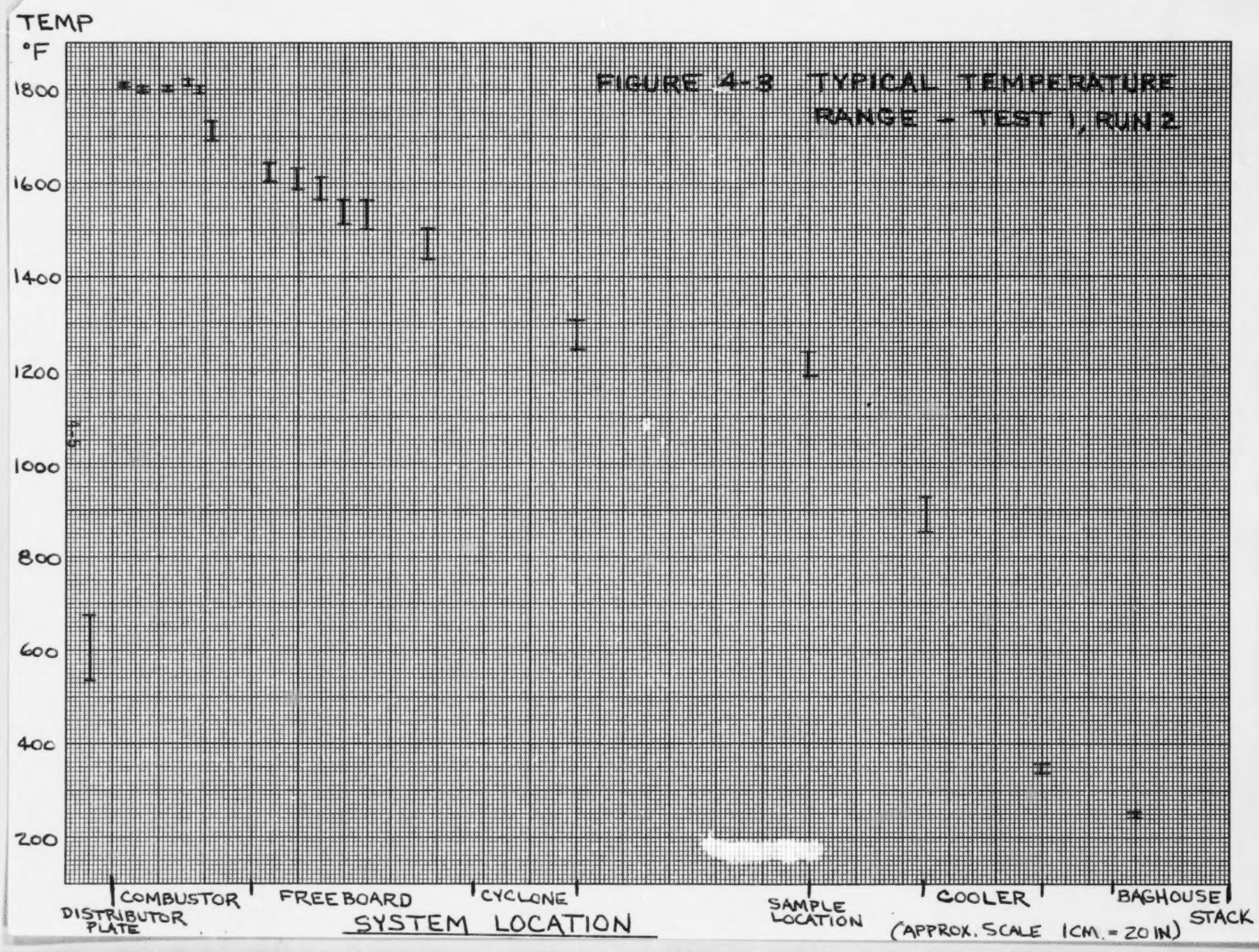


FIGURE 4-3 TYPICAL TEMPERATURE RANGE - TEST 1, RUN 2

total hydrocarbons difficult to accurately monitor. All carbon is assumed to be burned since little ash is present in the oil and excess air was high (>100%).

4.2 SAMPLING AND ANALYSIS RESULTS

4.2.1 VOST Results

Eight pairs of samples were taken over a continuous combustion run. Tenax and condensate results were averaged for the third, sixth, and eighth set of samples in order to evaluate the technique for typical sampler operation. Table 4-2 provides analysis results for each run in units of total milligrams per cubic meter. VOST samples were also taken in the sampling area and the fuel mixing shed. Results from these areas are given in Table 4-3.

Volatile compounds in the fuels were analyzed in the same way as the cartridges. The fuel volatile mixtures were spiked directly as the liquids onto Tenax cartridges and subsequently analyzed. Results for the fuels volatiles analysis are given in Table 4-4.

The large variation in volatiles found in the fuel can be attributed to several factors. Even though these compounds mixed satisfactorily with fuel oil in lab tests, it is possible that the fuel/waste mixture was heterogenous. Another explanation is that the concentrations changed during the sampling day due to the volatility of the test compounds. A further explanation may lie in the sampling method. The sample valve was located such that a small volume of tubing could trap fuel and retain it for some time. If the sample tube was not flushed sufficiently before each sample, the captured material may not accurately match the fuel/waste mixture. Analytical error could also account for some variability.

Table 4-2. VOST RESULTS FOR FLUIDIZED BED COMBUSTION SAMPLES

Set ID	Methyl Vinyl Ketone* (mg per cubic meter)	Tetrahydrofuran* (mg per cubic meter)
Run 1	0.0068	0.0243
Run 2	0.0064	0.0204
Run 3	0.0150**	0.0609**
Blank		ND

*Average results based on three Tenax cartridges only, carbon cartridges not quantifiable. Detection limits were 10 pg/DSCM for both compounds.
**Results based on only a single cartridge analysis

TABLE 4-3. VOST RESULTS FROM AMBIENT AIR SAMPLES (MG PER CUBIC METER)

Area	Methyl Vinyl Ketone	Tetrahydrofuran
Sampling Bay	None Found	0.05
Fuel Shed	0.04	0.15
Blanks	ND*	ND*

*Tenax-Carbon cartridges were used and no material was detected; however, high levels of background were encountered. Detection limit for area samples was 100 ng/DSCM.

TABLE 4-4. VOLATILES IN FUELS* (MG PER L FUEL)

Sample	Tetrahydrofuran	Methyl Vinyl Ketone
B139-A	7254.96	13576.5
B139-B	2179.63	23661.2
B139-C	4725.19	14934.2

*Three fuel oil samples were taken during the FBI burn. Although these samples were withdrawn at three different times during the day's run, no information was available or provided on the order of sample acquisition. Therefore, custody numbers were arbitrary and cannot be said to bear a relation to the actual sequence in which the samples were taken.

4.2.2 Glass SASS Results

Results from the analysis of the Glass SASS train samples are given in Table 4-5 (detection limits for the compounds sought are given in the table footnote).

Table 4-5. GLASS SASS TRAIN POHC ANALYSIS RESULTS (IN µg/DSCM)

Run #	Nitro-toluene	Chlorobenzo-trichloride	Isophorone	Triphenyl-methane
1	ND	ND	ND	0.553
2	ND	ND	ND	ND
3	ND	ND	ND	0.583

Note: ND = not detected. Detection limits for the POHC Compounds are as follows: chlorobenzotrichloride .5 µg/DSCM, Isophorone .3 µg/DSCM, Nitrotoluene .4 µg/DSCM, Triphenylmethane .3 ng/DSCM.

4.2.3 Dilution Tunnel Results

Results for the Dilution Tunnel samples analysis are provided in Table 4-6. Detection limits are given in the footnote to the table.

TABLE 4-6. DILUTION TUNNEL POHC ANALYSIS RESULTS (IN µg/DSCM)

Run	Nitro-toluene	Chlorobenzo-trichloride	Isophorone	Triphenyl-methane
1	ND	ND	0.50*	0.29
2	ND	ND	ND	0.42
3	3.45*	ND	ND	ND

*Results likely due to laboratory contamination since none of the semi-volatiles were found with the Glass SASS; therefore are not considered in DRE calculations.

Note: ND = no response for any of the indicator m/e POHC's. Quantitation limits for the POHC Compounds are as follows: chlorobenzotrichloride 0.21 µg/DSCM, Isophorone 0.13 µg/DSCM, Nitrotoluene 0.17 µg/DSCM, Triphenylmethane 0.13 µg/DSCM. Detection limits are two to ten times lower than the quantitation limits.

4.2.4 Fuels Analysis Results

Fuels were diluted and run under the same conditions as the Glass SASS samples. Results for the three fuel samples are given in Table 4-7.

TABLE 4-7. FUELS ANALYSIS RESULTS FOR GLASS SASS SAMPLING RUNS (mg/ml)

Run #	Nitro-toluene	Chlorobenzo-trichloride	Isophorone	Triphenyl-methane
1	7.10	1.95	3.24	4.36
2	6.02	3.07	3.89	5.86
3	3.61	.913	3.84	7.39

Approximately one kilogram of each surroge was added to approximately 25 gallons of fuel oil. The expected concentration of each compound in the mixture burned was ~10 mg/ml. These results show considerably smaller concentrations. The explanations for the precision and accuracy variances are the same as those for the VOST fuel results described in Section 4.2.1. These lower-than-expected concentrations were used in DRE calculations since they offer a worse case scenario.

4.2.5 Chloride Analysis

In addition to the organic compound analysis performed to determine destruction efficiency, chloride analysis was performed to determine the level of chloride emissions for environmental impact assessment. These analyses were also performed to evaluate the application of simultaneous sampling for organic materials and chloride and to evaluate the efficacy of the combined sampling and analytical procedures.

Methods

Chloride resulting from the combustion process was collected in the glass SASS in two places. The primary collection point was the condensate collected subsequent to the XAD-2 module. Any chloride carried past the condensate was collected in the impinger portions of the train. The first impinger was used as an additional condensate trap with no adsorbing solution, the second was loaded with a measured amount of deionized water (approximately 500 ml). The third impinger was loaded with 500 ml of 0.1-N sodium hydroxide.

A ten mililiter aliquot was taken from each of the sources of chloride in the SASS train. Each of these aliquotes was analyzed in triplicate by ion chromatographic techniques. Results of the total chloride found from each of the sampling runs is given in Table 4-8.

Table 4-8. Chloride Analyses from Glass SASS Samples (in mg)

Test Run	Total Chloride	Other Anions (qualitatively indicated)
1	**	
2	129.3	Nitrate, Sulfate, Organics
3	218.1	Nitrate

**Test Run 1, Condensate sample lost. Sampling probe not in stack properly. Results not reported due to sampling problems.

4.2.6 Analytical Quality Control

The quality control procedures applied to this effort are described in the approved quality assurance plan (included as an appendix of this report) titled: Fluidized Bed Combustion/Incineration of Surrogate Waste dated 3/1/83, and subsequent modification dated 12/7/83. Pertinent data and any noted unusual events are provided in the QC report which follows. Copies of sample custody sheets are attached as part of this QC report.

4.2.6.1 VOST Quality Summary

Results for individual cartridge analyses are given in Table 4-9. Results of the individual condensate sample analyses are given in Table 4-10. Table 4-11 contains the recovery factors for methylfuran spiked onto the VOST cartridges containing the source samples. Precision determination for the actual source samples can not be performed since each sample is a single point and no analytical replicates were possible. The average result reported for the VOST samples and the associated precision can be calculated. The result of this

TABLE 4-9. RESULTS OF INDIVIDUAL CARTRIDGE ANALYSIS
(in total ng per cartridge)

Sample Number	Cartridge Number	Methyl Vinyl Ketone	Tetrahydrofuran
B118 A	1-1-1	126.5	54
B118 A	1-1-2	***	***
B118 B	1-2-1	183	1208
B118 B	1-2-2	***	***
B118 C	1-3-1	108	228
B118 C	1-3-2	143**	222**
B119 A	2-1-1	89	497
B119 A	2-1-2	***	***
B119 B	2-2-1	125	184
B119 B	2-2-2	***	***
B119 C	2-3-1	179	568
B119 C	2-3-2	246	414
B120 A	3-1-1	*	*
B120 A	3-1-2	***	***
B120 B	3-2-1	305	1236
B120 B	3-2-2	***	***
B121 A	Blank****	***	***
B122 A	Area 1	ND	113.5
B122 B	Area 2	80	326.0

* Data lost, GC/MS data system malfunction

** Compounds of interest were detected, however, detection limit and accuracy suffer because peaks eluted on the tail of a high concentration unknown compound.

*** Peaks of interest not detectable, detection limit unknown due to tailing from an unknown component.

**** Control cartridges both containing Tenax/charcoal were left to ambient air at the sampling site during the final 30 minutes of the VOST run.

Area 1 is the monitoring sample for the sampling port area.
Area 2 is an area monitoring sample for the fuel mixing shed. Cartridge number consists of test no., run no., and cartridge designation.

precision evaluation is given in Table 4-12 and reflects the total source, sampling, and analysis variability.

TABLE 4-10. RESULTS OF VOLATILES ANALYSIS FROM VOST CONDENSATE (in total ng per cartridge)

Sample	Run Number	Methyl Vinyl	Tetrahydrofuran
B118 D	1	ND	ND
B119 D	2	ND	ND
B120 C	3	ND	ND

ND indicates no compound detected.
Quantitation limit for both compounds is 3 pg/DSCM.

TABLE 4-11. INTERNAL RECOVERY STANDARDS FACTORS FOR VOST CARTRIDGES

Sample	Run Number	Factor
B118 A	1-1	2.22
B118 B	1-2	1.39
B118 C	1-3	*
B119 A	2-1	1.10
B119 B	2-2	2.32
B119 C	2-3	3.20
B120 A	3-1	**
B120 B	3-2	1.40

*No internal standard injected.
**Data system malfunction caused complete data loss.
Recovery factor = actual source concentration ÷ observed concentration.

TABLE 4-12. QUALITY OBJECTIVES AND OBJECTIVE ACHIEVEMENT SUMMARY FOR VOST ANALYSES (GIVEN IN RELATIVE STANDARD DEVIATION)

Compound	Stated Precision Objective	Achieved Precision
Methyl Vinyl Ketone	50-150%	52%
Tetrahydrofuran	50-150%	47%

4.2.6.2 SASS Quality Summary

Table 4-13 summarizes the quality objectives and achievements for the SASS sampling and analyses. In order to meet the goals for data quality, a precision within 32% was necessary with at least two of the planned three samples required. All quality objectives were met except for triphenyl methane. The percent coefficient of variation for this compound falls outside the range originally established. Two of the results from runs of the SASS were quite similar (0.553 and 0.582 μg/DSCM). The third measurement (0.00) provided the most significant variation, yet application of the student t-test (at the 95% inclusion level) indicated a range for 3 data points from -0.087 to 0.843. This range includes all the data points recorded, therefore, the percent coefficient of variation is reported as 87%.

The precision measurements here include the total variation from all sources. Possible sources of variation include fuel handling, combustor operation, flue gas sampling, sample recovery, and sample analysis.

TABLE 4-13. SASS QUALITY OBJECTIVES AND OBJECTIVE ACHIEVEMENT SUMMARY

Compound	Stated Precision	Stated Completeness	Achieved Precision	Achieved Completeness
4-Nitrotoluene	32%	66%	0%	100%
Chlorobenzo-trichloride	32%	66%	0%	100%
Isophorone	32%	66%	0%	100%
Triphenylmethane	32%	66%	87%**	100%

Included for completeness are Tables 4-14 through 4-16 of analytical results and calibration data for SASS samples.

**Triphenylmethane solids were found as a residue in the fuel oil tank. High variation in the measurement of surrogate wastes in the fuel may be the result of settling or volatilization of the test compounds and may explain the high variation in the analytical results.

Table 4-14. GCMS ANALYTICAL RESULTS: GLASS SASS SAMPLES

TSS ID	DESCRIPTION	Total.Micrograms Found				
		D_{10} ANTHRACENE (240μg spiked)	NITROTOLUENE	CHLOROBENZOTRICHLORIDE	ISOPHORONE	TRIPHENYL METHANE
B106B	XAD Extract, Run # 1	150.82	0.0	21.65	0.0	16.14
B 114 F	XAD Extract Run # 2	0.0 *	0.0	0.0	0.0	0.0
B116B	XAD extract, Run # 3	0.0 *	0.0	0.0	0.0	7.84
B106G	XAD-2 Blank	288.01	0.0	0.0	0.0	0.0
B116G	XAD-2 Blank	247.02	0.0	0.0	0.0	0.0
B105AB	Dilution Tunnel Filter, Run # 1	4.239	0.0	0.0	1.743	1.007
B105CD	Dilution Tunnel Filter, Run # 2	23.693	0.0	0.0	0.0	3.030
B105 E	Dilution Tunnel Filter, run # 3	250.500	25.640	0.0	0.0	0.0
B105GI	Dilution Tunnel Filter Blank	0.0**	0.0	0.0	0.0	0.0
B106C	Condensate Run # 1	SAMPLE LOST				
B 116C	Condensate, Run # 2	5.930	0.0	0.0	0.0	0.0
B114B	Condensate, Run # 3	160.660	0.0	0.0	0.0	6.520
B116AE	Filter Extract & Probe Rinse Run # 2	97.330	0.0	0.0	0.0	8.660

* decomposition of D_{10} anthracene to D_{10} dibenzoquinone
**none spiked

Table 4-15. CORRECTION FACTORS (INTERNAL STANDARD: DIBROMOBENZENE)

Sample	Description	Correction Factor
B106B	XAD Extract Run #1	x 3.82
B114F	XAD Extract Run #2	x 1.24
B116B	XAD Extract Run #3	x 1.2
B106G	XAD-2 Blank	x 0.76
B116G	XAD-2 Blank	x 1.47
B105AB	Dilution Tunnel Filter, Run #1	x 1.87
B105CD	Dilution Tunnel Filter, Run #2	x 1.16
B105E	Dilution Tunnel Filter, Run #3	x 5.9
B105GI	Dilution Tunnel Filter Blank	_____*
B106C	Condensate Run #1	Sample Lost
B116C	Condensate Run #2	x 1.2
B114B	Condensate Run #3	x 2.03
B116AE	Filter Extract	x 2.1

*No Dibromobenzene Injected

Correction factors represent the ratio of expected dibromobenzene in the sample to that actually found. These factors allow the operator to compensate for instrument response change from day to day.

TABLE 4-16. LINEAR REGRESSION INFORMATION

D_{10} Anthracene
Slope: 11.41, Intercept: +451.31,
Correlation: 1.00

Triphenyl Methane
Slope: 35.19, Intercept: - 283.8,
Correlation: 0.99

Nitrotoluene
Slope: 17.26, Intercept: - 605.6,
Correlation: 0.87

Isophorone
Slope: 19.9, Intercept: - 178.99,
Correlation: 0.99

Chlorobenzotrichloride
Slope: 20.67, Intercept: - 180.49,
Correlation: 1.00

Note: Typical Abundance Counts are in the 1000-10,000 range

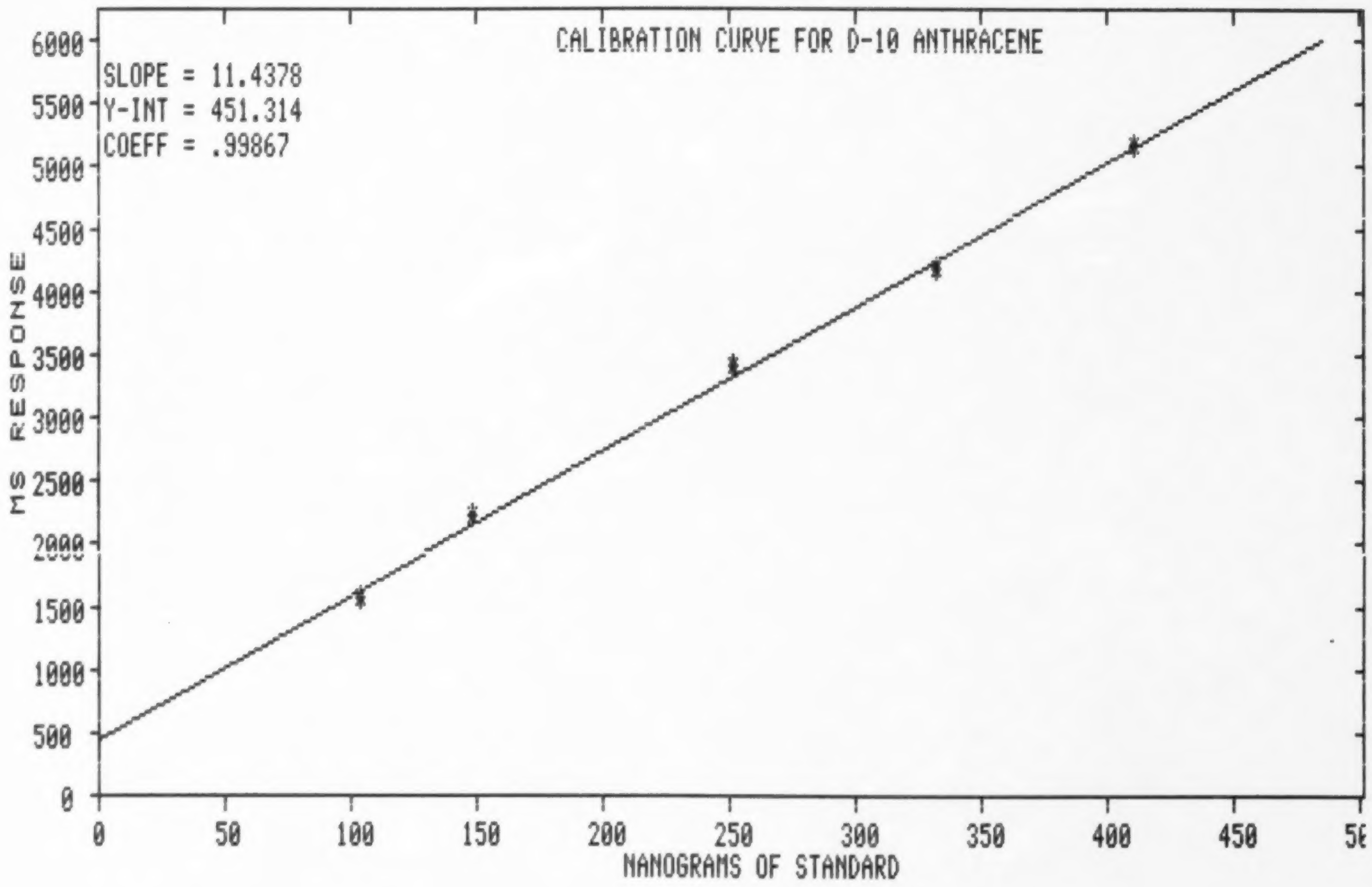


Figure 4-4. Calibration Curve for D-10 Anthracene.

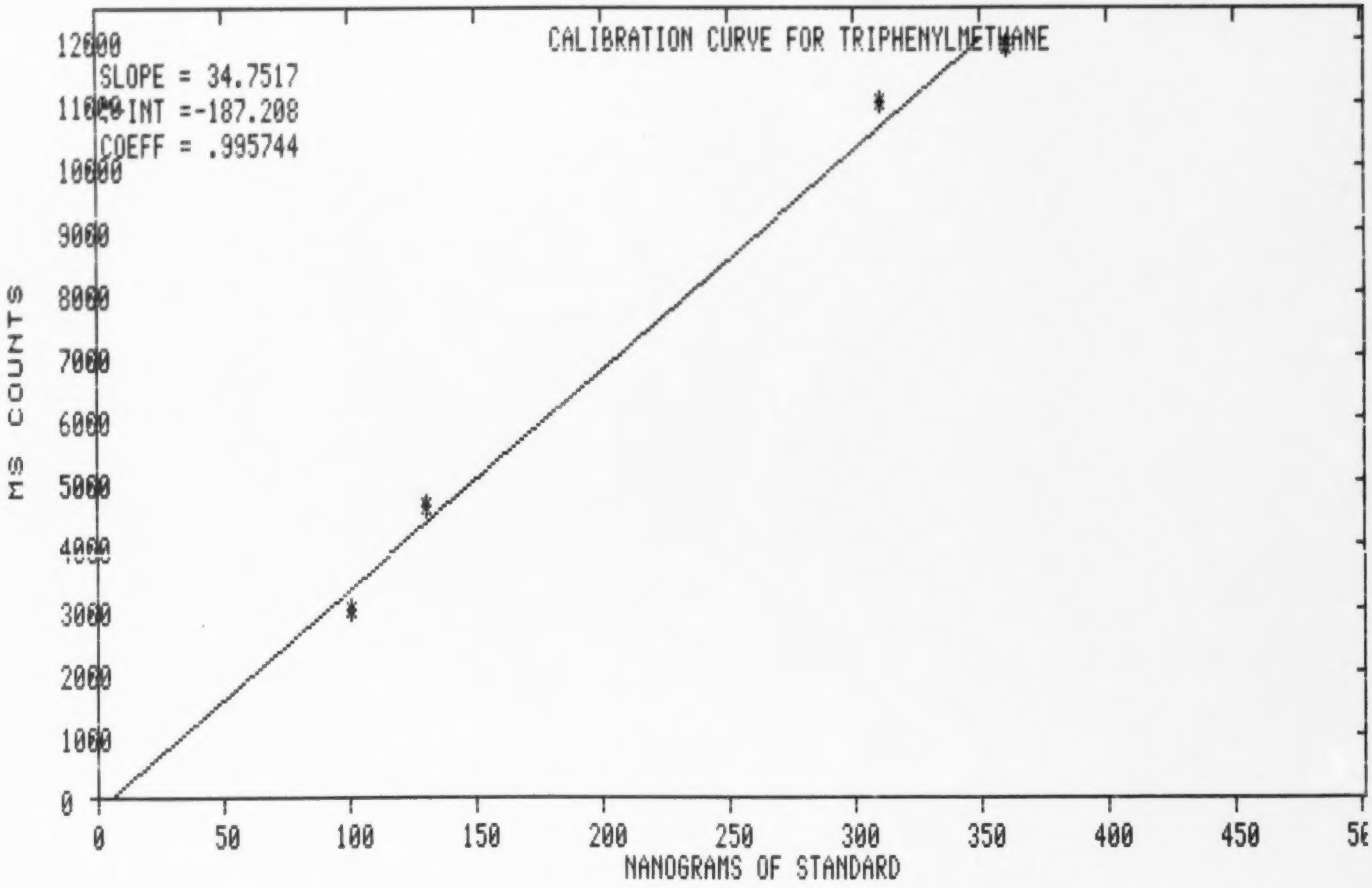


Figure 4-5. Calibration Curve for Triphenylmethane.

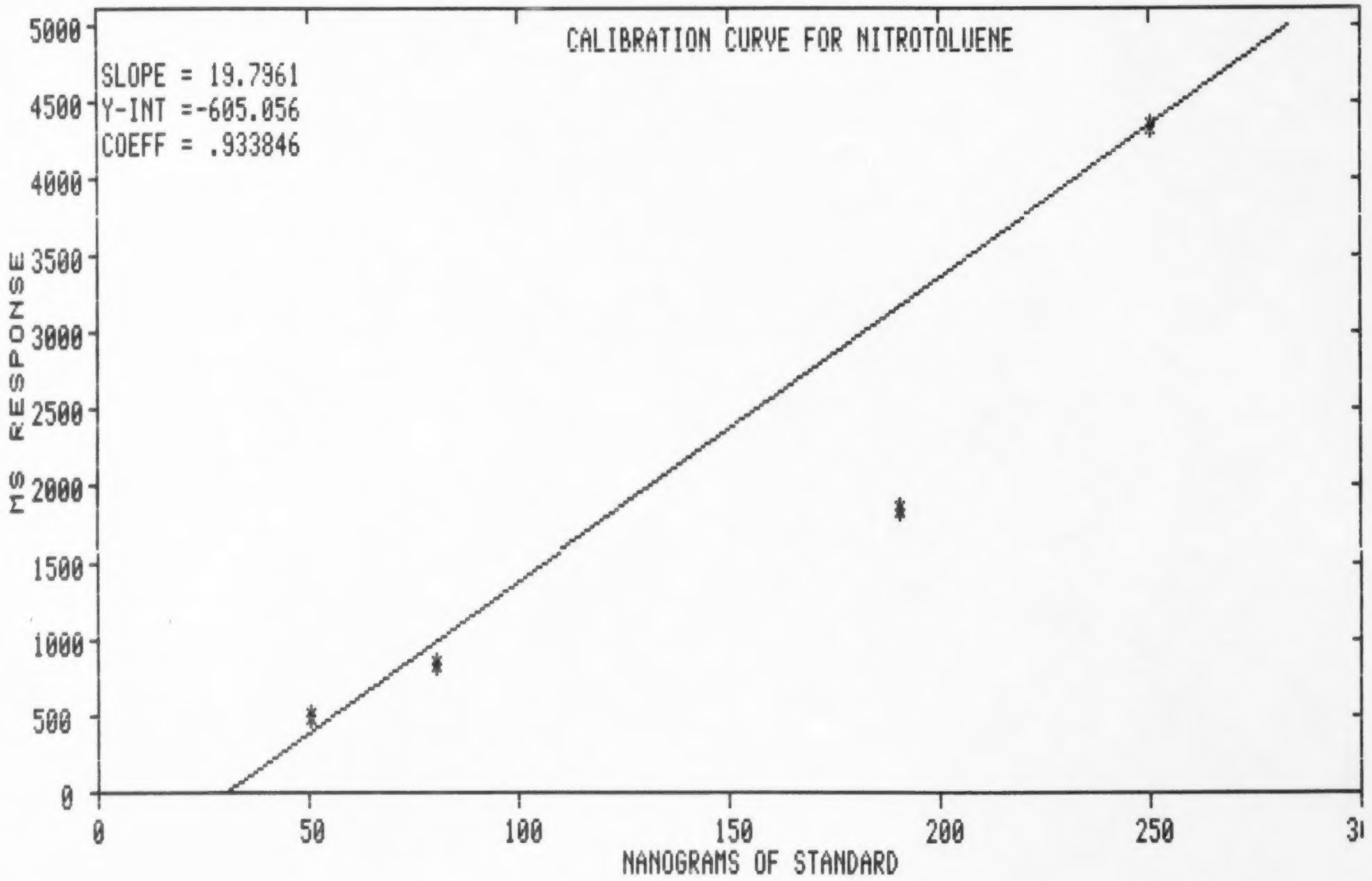


Figure 4-6. Calibration Curve for Nitrotoluene.

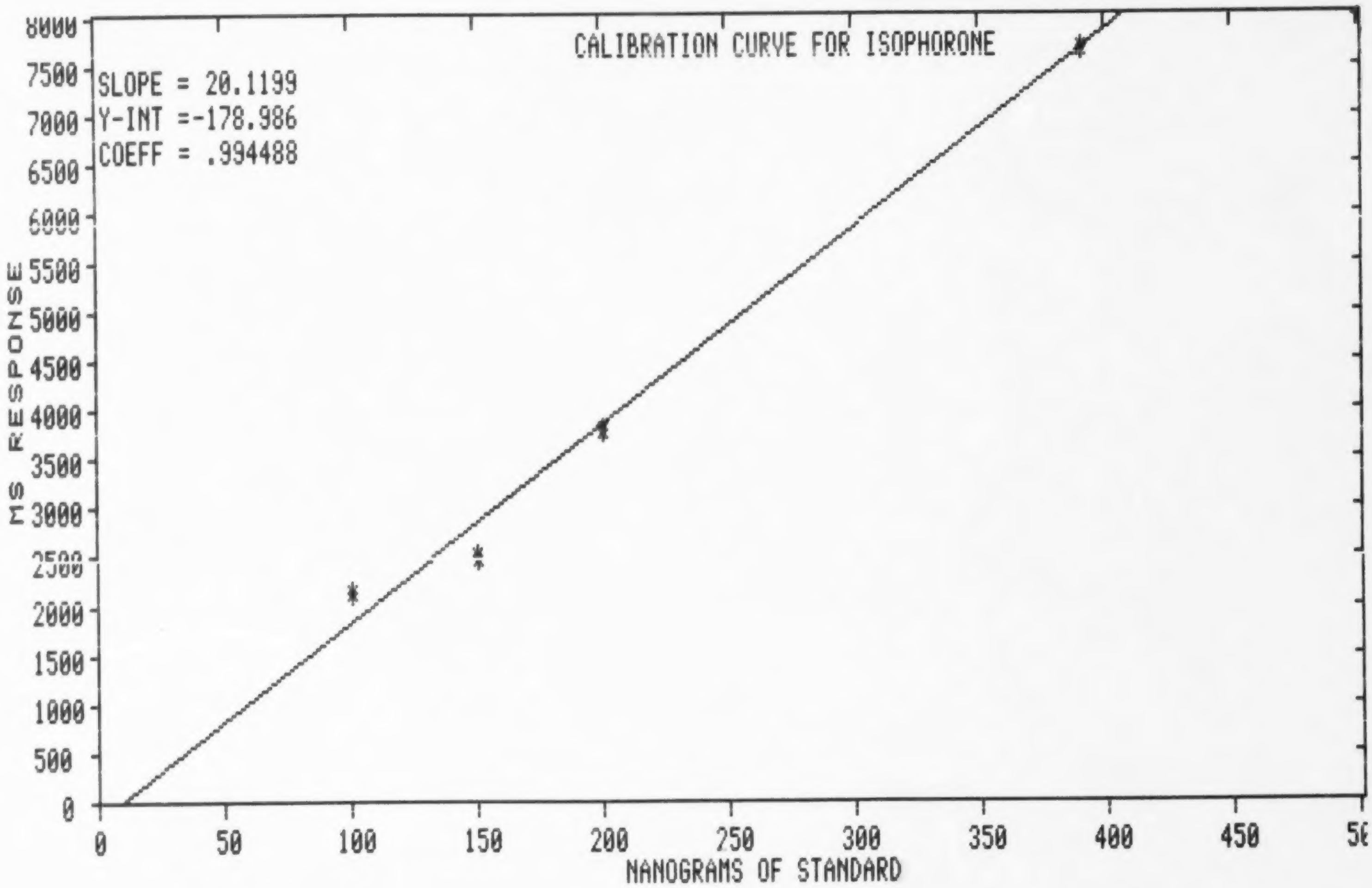


Figure 4-7. Calibration Curve for Isophorone.

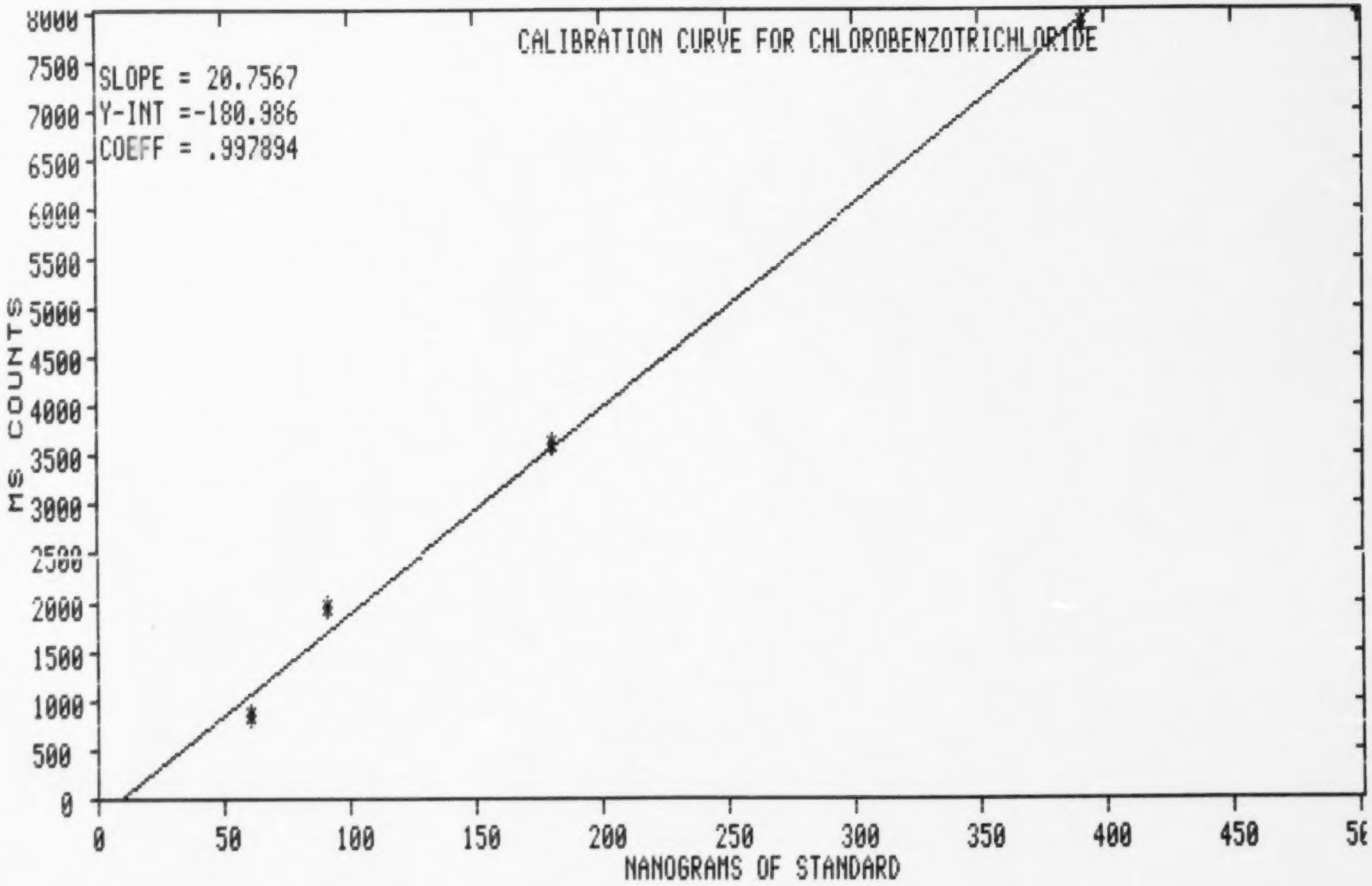


Figure 4-8. Calibration Curve for Chlorobenzotrichloride.

4.2.6.3 Dilution Tunnel Quality Summary

Table 4-17 summarizes the quality objectives and achievements for the Dilution Tunnel results. Results for all of the semivolatile compounds are given in this table for completeness even though laboratory tests indicated that only the triphenyl methane would be retained on filters at ambient temperatures. Results for the semivolatiles (nitrobenzene, chlorobenzotrichloride, and isophorone) are most likely due to laboratory contamination. The dilution tunnel was not designed for semi-volatile organic compound collection, and independent laboratory validation (EPA Contract 68-02-3118) showed no recovery for the semi-volatile compounds used in this study. Therefore, the results from the dilution tunnel for semivolatile constituents should not be considered indicative of the destruction efficiency of these compounds combusted in the Fluidized Bed Incinerator. Results from the dilution tunnel fall outside the originally established goals for precision; however, the results for triphenyl methane may be useful for destruction efficiency calculations if the levels are low enough to demonstrate 99.99% within the error of the measurement.

TABLE 4-17. QUALITY OBJECTIVES AND OBJECTIVE ACHIEVEMENT SUMMARY FOR DILUTION TUNNEL RESULTS

Compound	Objectives Precision	Objectives Completeness	Achieved Precision	Achieved Completeness
Nitrotoluene	32%	66%	173%	100%
Chlorobenzotrichloride	32%	66%	0%	100%
Isophorone	32%	66%	347%	100%
Triphenylmethane	32%	66%	91%	100%

4.2.6.4 XAD-2 Resin Quality Control Documentation

XAD-2 was prepared according to Level 1 methodology and quality control procedures, including residual methylene chloride, residual extractable organics and residual gravimetric analyses were performed. None of the compounds spiked into the fuel as surrogate wastes were found in the XAD-2 resin blanks. Table 4-18 provides the standard QC documentation for the XAD-2 used in these tests.

TABLE 4-18. XAD-2 QA/QC RESULTS FOR COMBINED BATCH B101A AND B101B

	Residual Methylene Chloride μg/g	Residual TCO μg/g	Residual Gravimetric μg/g
Replicate 1	111.6	ND	77.7
Replicate 2	76.5	ND	ND
Average	94.1	ND	77.7
QC Limit	1000.0	10	25

ND indicates not detectable at the detection limit of 39 μg/g.
None of the surrogate compounds were detected in the XAD-2.

4.3 DESTRUCTION AND REMOVAL EFFICIENCY RESULTS

Data obtained from the EPA Technical Support Staff provided the basis for calculating Destruction and Removal Efficiency (DRE) values for each test compound using the facility at the specified run conditions. These results are tabulated in Table 4-19. The DRE is calculated per the following equation:

$$\mathrm{DRE} = \frac{\text{waste in} - \text{waste out}}{\text{waste in}} \times 100$$

TABLE 4-19. DESTRUCTION AND REMOVAL EFFICIENCIES

Test Compound	Method (Train/Anal)	Test/ Run		Input Concen. (mg/m^3)	Output Concen. (mg/m^3)	DRE
Tripheylmethane	SDSS/GC/MS	1/2		322.39	.00042	99.99987
	Glass SASS/GC/MS	1/3		374.82	.000583	99.99984
Nitrotoluene	Glass SASS/GC/MS	1/2		331.19	ND	99.9998+
		1/3		183.10	"	99.9997+
Isophorone	Glass SASS/GC/MS	1/2		219.51	"	99.9998+
		1/3		194.76	"	99.9998+
Tetrachlorotoluene	Glass SASS/GC/MS	1/2		168.90	"	99.9997+
		1/3		46.31	"	99.9989+
Tetrahydrofuran	VOST/GC/MS	2/1		484.96*	.0243	99.9886**
		2/2		145.70	.0204	
		2/3		315.86	.0609	
			avg	315.51	.0359	
Methyl Vinyl Ketone	VOST/GC/MS	2/1		907.52*	.0068	99.9992**
		2/2		1581.64	.0064	
		2/3		998.28	.0150	
			avg	1162.48	.0094	

*Unknown order

**Based on average inlet and outlet

***Detection limits:

***Detection limits:	Nitrotoluene	0.4 µg/DSCM
	Isophorone	0.3 µg/DSCM
	Tetrachlorotoluene	0.5 µg/DSCM

Calculated DRE's based on these minimum limits.

4.4 FACILITY OPERATION EVALUATION

As previously described, the major facility operational concern occurred during start-up when the blower motor was overloaded. Once bed temperatures reached design steady state operation levels, the facility performed well.

One problem caused the premature shutdown of the facility. During Test 1 - Run 1, the first oil feed nozzle plugged completely after gradually requiring larger valve openings and higher supply pressures to maintain flowrate. A 60-micron in-line filter was partially plugged with a gritty material, likely dirt or filings from the reused plumbing components. The oil nozzle itself was plugged with a black flaky material which appeared to break loose from curved walls of the tubing or instrumentation. Tests conducted by EPA's Technical Support Staff verified that the material was not any of the test compounds because it was insoluble in acetone or methylene chloride. The most likely source was the flow totalizer found in the flow control module used in the TVA - Widow's Creek PCB Test Burn. The flow totalizer, oversized and unnecessary for this run, was removed for future runs.

Non-critical observations made during the operation or from operation data are discussed in Section 6-1, Facility Conclusions and Recommendations.

4.5 POST-TEST FACILITY EVALUATION

The particle size evaluation of the bed material indicates that erosion of bed material occurred through the approximate 66 hrs of facility operation. Table 4-20 shows the particle sizing results of the bed material before and after testing, indicating a 10.8% reduction in larger beads. An examination of the combustor section indicated that the grinding effect of the fluidized bed had little effect on the refractory surfaces but several cracks were found likely due to excessive temperature gradients experienced near the gas burner inlet during refractory curing and facility start-up.

Table 4-20. Bed Material Particle Sizing

Sample	Sieve No (in Ø)	As Received				Post-Test - 66 Operational Hours				Change
		Total Weight (gm)	Tare Weight (gm)	Collected Weight (gm)	Percent	Total Weight (gm)	Tare Weight (gm)	Collected Weight (gm)	Percent	
1	16 (.0469)	1020.8	746.5	274.3	15.7	779.7	746.5	33.2	3.2	-12.5
	18 (.0394)	1890.1	733.2	1246.9	71.4	1584.8	733.2	851.6	81.3	+9.9
	20 (.0331)	981.0	763.5	224.5	12.9	926.4	763.5	162.9	15.5	+2.6
	30 (.0234)	715.3	715.7	-	-	715.4	715.4	-	-	-
2	16 (.0469)	929.1	745.6	183.5	11.3	798.3	745.6	52.7	3.2	-8.1
	18 (.0394)	1857.4	732.3	1125.1	69.2	1996.4	732.3	1264.1	76.9	+7.7
	20 (.0331)	1078.9	761.9	317.0	19.5	1088.6	761.9	326.7	19.8	+0.3
	30 (.0234)	-	-	-	-	-	-	-	-	-
Total	16 (.0469)	1949.9	1492.1	457.8	14.0	1578.0	1492.1	85.9	3.2	-10.8
	18 (.0394)	3747.5	1465.5	2282.0	69.7	3581.2	1465.5	2215.7	78.9	+9.2
	20 (.0331)	2059.9	1525.4	534.5	16.3	2007.0	1525.4	481.6	17.9	+1.6

Little particulate was found in the cyclone catch as expected. The few grams of material were primarily loose material in the cyclone from previous operations. It was known that the cyclone refractory was in poor condition prior to the tests.

A larger volume of particulate, (<1 inch depth in the catch drum) was found in the baghouse catch. Again, the bulk of the material was likely reentrained particles from previous coal-burning operations. An additional quantity of material was found in the cooling tube drawer.

Samples of the solid materials (bed material, particulate catches) were analyzed by TRW, Inc. to determine the presence of any of the test compounds. All samples were found to be negative, i.e. levels of all test wastes were below the analytical detectability level of 15 ng/g. These results support the high destruction efficiencies.

4.6 WIPE SAMPLES

A list of the wipe samples taken and results can be found in Table 4-21. The majority of samples were taken from spill areas or known contaminated equipment before and after clean up procedures. The spills and clean up procedures and findings are discussed in Section 5, the Health and Safety Report. Additional wipe sample information was transmitted during the test and post-test period and is found in Appendix C.

4.7 ENVIRONMENTAL ASSESSMENT

The destruction and removal efficiencies for the test compounds verify that negligible quantities were released through the exhaust stack. The actual emission rates are probably less than the DRE's suggest since the sampling was performed upstream of the flue gas cooler, baghouse and draft fan. Therefore, the calculated DRE's are actually destruction efficiencies with the "removal" component of the DRE occurring downstream of the sampling location. As stated in

Table 4-21. Wipe Sample Results

Spill	Sample No.	Sampling Location	Possible Presence of Triphenylmethane
A	B109D	Wipe after cleanup under regulator	Yes
-	B109E	Fuel feed line rinse	Yes
B	B112B	Wipe in front of FBC	No
C	B112C	Wipe fuel, supply module	Yes
A	B112D	Wipe under valve	Yes
D	B112E	Wipe fuel feed box	Yes
-	4770	Cleaned 'contaminated' wrench	No
-	4771	Waste receptacle top	No
-	4772	Sampling port on stack	No
-	4773	Temperature probes	No
-	4774	Recycle line	Yes (170 $\mu g/ft^2$)
-	4775	Top of drum, clean fuel	Yes (12.5 $\mu g/ft^2$)
-	4776	Regulator on vent	No
-	4777	Inside of red flue gas line from cooler	No
-	4778	Probe tip nozzle	No
-	4779	Top of feed barrel	Yes (330 $\mu g/ft^2$)
-	4780	Cooling water condensate	No
-	4781	Floor wipe at Bay Door to outside	No
-	4782	Area A	No
-	4783	Area C	Yes (2.53 $\mu g/ft^2$)
-	4792	Top of feed barrel after cleaning	Yes (83 $\mu g/ft^2$)
-	4793	Fuel shed walls	No

the Test Plan (Volume I) the primary hazardous constituent of concern was HCl since it would be the result of effective destruction of the tetrachlorotoluene. The operating parameters were considerably different from those predicted in the Test Plan. The fluidization velocity was ~ 3.3 ft/sec for Test 2 rather than the proposed 2 ft/sec. This resulted in increased flowrates from the predicted 31.8 scfm to ~ 56 scfm, and an increase in fuel consumption from the expected 0.6 gal/h to rates ranging from 1.1 to 1.4 gal/hr. The theoretical HCl emission rate, assuming 100% of the chlorine was freed to form HCl, would have been 26.27 gm/hr or a concentration of 276 mg/scm. This theoretical value would produce slightly higher worst case concentrations than predicted on the test plans (based on 218 mg/scm) but still well below unsafe limits. The chloride sampling and analyses from the glass SASS runs indicate the following results:

TABLE 4-22. STACK CHLORIDE CONCENTRATIONS

Test 2 Run	Waste Conc. in fuel (mg/ml)	Input Waste Conc. (mg/dscm)	Input Chlorine (mg/dscm)	Potential HCl (mg/dscm)	Outlet Total Chloride (mg)	Outlet Sample Size (dscm)	Outlet Conc. (mg/dscm)
1							
2	3.07	168.90	104.20	107.16	129.3	30.46	4.25
3	.913	46.31	28.57	29.39	218.1	31.30	6.97

These results indicate that any encountered concentrations of HCl in the work area fall at least two orders of magnitude below the ACGIA eight-hour exposure limit of 7000 $\mu g/m^3$, therefore, providing no health hazard to area personnel.

The actual emission rates were probably less than the values shown in Table 4-22 due to the low stack temperature. Since HCl is highly soluble in water and the stack temperature was less than 200°F for all runs, the condensation on the stack and draft fan surfaces would be a hydrochloric acid solution.

The analysis of personnel monitoring badges showed that none of the organic compounds tested were present in the work area. The badges worn by personnel mixing the volatile compounds in the fuel shed indicated positive results as expected, but these personnel were wearing organic and acid gas respirators while in the fuel shed. Likewise, the area monitor in the work area showed no detectable quantities of MVK but did detect .05 mg/m^3 tetrahydrofuran. The detected level of .017 ppm is four orders of magnitude below the TVL-TWA of 200 ppm. The monitor from the fuel shed indicated .04 mg/m^3 MVK and 0.15 mg/m^3 tetrahydrofuran. The facility was operated at negative pressures downstream of the combustion zone so that any harmful constituents in the flue gas could not escape into the work area. The combustion zone is at a slight positive pressure but all taps or openings in the combustor section were sealed and leak-checked prior to testing. The most likely explanation for the tetrahydrofuran in the work area is that it drifted from the fuel shed and was drawn into the Wing G air intake and distributed through the air handling system.

There were small spills of the fuel/waste mixture in the work area which occurred during or after an upset condition. The fuel nozzle plugged during Test 1, Run 1 requiring facility shutdown prior to flushing the fuel feed several drops of fuel/waste mixture were spilled on the concrete floor. The plugging also caused a high pressure (~60 psi) to build up in the fuel feed line. When the oil shut-off valve was closed the air nozzle purge valve opened subjecting the air line to the high pressure. The purge air regulator, set at 15 psi, vented the excess pressure into the work area causing some fuel/waste mixture to drip on the floor. These were the only full concentration fuel/waste spills on the concrete floor. Other spills occurred in the fuel feed system modules or on drum lids in the fuel handling shed. One troublesome spill followed a spill in the flow control module during the removal of the flow totalizer after Test 1, Run 1. The silicone sealant used in the plexiglass module was unsuitable for fuel oil and solvents. Therefore, the fuel/waste mixture and cleanup solvent were not contained in the module but dripped undetected for a short time onto the floor.

All of the spills indicated in Table 5-2 consisted of amounts ranging from 1 drop to <10 ml of fuel/waste mixture resulting in low levels of fugitive test system. As a result, when the system was dismantled to clean the nozzle, compounds (<0.2 mg) before cleanup efforts. Therefore, no harmful effects to personnel resulted from the spills.

5.0 HEALTH AND SAFETY PROGRAM

5.1 PURPOSE AND SCOPE

A major objective of this project was the maintenance and operation of test facilities in an exemplary manner. A health and safety plan (see Appendix A) was designed to implement, as part of this project, a state-of-the-art health, safety, and monitoring and analysis procedures. The overall plan included developmental work with seminar presentations and documenting established safety protocols. The purpose of this section is to report on the effectiveness of a standardized plan which was developed to insure the health and safety of personnel participating in the fluidized bed incineration (FBI) project at the AEERL facility.

This section of the overall Phase I report will present the health and safety protocols developed, with observations made during Phase I of the project. The discussion presented will elaborate on the observed difficulties with protocols, facility limitations, project scheduling, analytical results, and observations on the details of personnel performance. Items for further consideration will be suggested for future protocol improvements, methodology adaptions, and facility refinements. Due to the inherent confinement of the fluidized bed combustion facility (FBC), and required interaction of Acurex and TSS personnel, a thorough understanding of the procedures in the overall health and safety plan and training manual is considered necessary to adequately understand the problems encountered with the Phase I program.

The Phase I test schedule included four test days within two test periods. A test period included the mixing of selected test compounds, test runs with sampling trains, and maintenance/cleanup activities.

The first test period began on May 5, 1983 with the mixing of the group of selected test compounds classified as semi-volatiles. The compounds mixed were nitrotoluene, triphenylmethane, isophorone, and tetrachlorotoluene. Two sample collection methods were used for data collection during this test period. There were a Source Assessment Sampling System (glass SASS) with a glass XAD-2 resin module assembly for measuring semi-volative organic constituents and a Stack Dilution Sampling System (SDSS) for measuring the condensible organic detection. The test runs on May 5, 1983 were aborted due to facility problems. Some clean-up and maintenance were required on May 5, 1983 and the next two non-testing days, May 6 and 9. The test runs for this period were completed on test days May 10 and 12 with May 11 used for facility maintenance. Spill clean-ups were completed on May 13 to complete the end of Phase I of the project.

The second test period began on May 17, 1983 with the mixing of the test compounds in the volatile class. The selected compounds included tetrahydrofuran and methyl vinyl ketone. A Volatile Organic Sample Train (VOST) was the sample collection method used for data collection and the test runs were completed on May 17. Maintenance and spill clean-up activities were performed from May 18 to May 24.

5.2 HEALTH AND SAFETY PROCEDURES

The FBC Health and Safety Training Seminar Manual which prescribed the initial procedures for implementing the health and safety plan for this project and the Health and Safety Manual for the U.S. Environmental Protection Fluidized Bed Combustion Test Facility Work Plan should be referred to as background (Appendix A of this report). TRW was assigned the responsibility to monitor Phase I conformance with health and safety protocols to assist in refining and improving safety procedures and routines. A method of observing project activities during Phase I was designed. The method included developing a checklist for observing adherence to the provisions of the Health and Safety Plan, documenting discrepancies and discussions providing feedback and opportunity of possible improvements from project personnel. Improvements included development of a spill chart/log, improved dress procedures, and a detailed spill clean-up procedures.

The checklist form was devised as a means for monitoring the activities at the FBC facility. The checklist monitored the personnel activities and facility conditions in the four areas of the FBC facility: (1) fuel feed shed, (2) facility operation, (3) sampling area, and (4) overall facility observation. The FBC Phase I Health and Safety Checklist is present in Appendix A. The rating system utilized for each item on the checklist was on a 1-3 scale. A rating of 3 indicated an acceptable level within the context of the prescribed heath and safety protocol or mutually approved procedures, and therefore no comment was required. A rating of 2 indicated a marginally acceptable practice but required comment and action to modify the practices as necessary. A rating of 1 indicated that the level of performance was unacceptable and action was required to change either personnel or facility practices.

The sequence for monitoring the FBC activities with the checklist was achieved with two designated independent health and safety observers: (1) the TRW Safety Monitor and (2) the Acurex Health and Safety Project Monitor. This monitoring procedure was approved by the EPA/RTP Health and Safety Office. Timing of health and safety observations was arranged to be non-concurrent for the purpose of producing a larger data set. The TRW monitor performed observation during the initial, mid-point, and final stages of each test day. These observations coincided with start-up and recovery of the source samples and the collection of three fuel feed samples during each day of testing. The Acurex monitor performed observations generally during times when sampling activities were not occurring except for when an initial observation each test day which was made concurrently with the other observer in order to determine consistency between the two observers. The completed checklist forms were filed on a daily basis and are stored in the project file. Further documentation was maintained by the TRW safety monitor for items not on the original checklist which required additional input. Improvements resulting from health and safety monitoring included designation of a dress/undress area, developing and implementing a detailed dress procedures, developing and implementing a clean-up procedure for potentially contaminated spills, and maintaining a spill chart/log with a detailed schematic of spill sites.

The dress area included a clean area for donning new safety outfits and doffing area with disposal containers for shedding and properly disposing of contaminated outfits. The dress procedure was developed for the correct sequence of donning and shedding the safety outfits. The dress procedure is presented in Appendix B.

Process spills occurred during initial Phase I facility operation and during facility maintenance. Five such spills was identified and treated as potentially contaminated areas. The initial spill (A) occurred when the probe nozzle plugged (5/5/83) and the pressure buildup caused the spiked fuel oil to spill out through a feed line regulator onto the facility floor. The liquid spilled contained the five semi-volatiles mixed in fuel oil. The nature of the known contaminated spill required spill containment and decontamination procedures. A second spill (B) occurred (5/6/83) while performing maintenance to unclog the probe. This spill was believed to be acetone used in flushing probe and analysis showed negative results. Two spills (C and D) occurred at the fuel feed module when a disconnected feed line leaked spiked fuel into the feed module (5/10/83). Spill C was the floor area under the Northwest corner of the module that the fuel leaked from. Spill D was the inside of the feed module. The liquid spilled contained the five semi-volatiles mixed in fuel oil. The liquid leaking from the module was cleaned immediately; while the module inside was cleaned after the test was completed. An oversight of the SASS testing crew caused some problems with maintaining the integrity of the area. The SASS cooling water was dumped from the mezzanine area into the facility operational area. The water was not contaminated but the presence of the liquid in this area caused problems maintaining a spill proof area. Another spill was found when the condensate trap on the sample line for the continuous process monitors flooded (Spill E). The process gas was being drawn through the continuous monitors while burning the five semi-volatiles. Therefore, this area was treated as potentially contaminated. Another spill was in the floor area where the tools or equipment were air dryed (Spill F). One spill area was added after the completion of testing. The top of the fuel tank in the shed (Spill G) was analyzed and produced positive evidence of contamination. Various spills could have occurred from both test periods during mixing or sampling activities. Therefore, the semi-volatiles and volatiles compounds tested caused this area to be treated as a potentially contaminated area.

The health and safety protocols provided guidance for decontamination and sampling of identified spill areas. During the project test period the five areas were identified as potentially contaminated. The details are discussed further in the subsection presenting analytical results. Due to the fact that even after clean-up, certain spill areas remained contaminated, a revision of the decontamination protocols was made. The revised decontamination procedures was employed on the spill areas with positive analytical results and the results of subsequent analytical screening are presented in Section 5.4. Appendix B presents the revised decontamination procedures.

A spill chart and log was developed for recording pertinent information pertaining to the spill areas. Location of the spill, clean-up procedures, and analytical results were recorded for each spill site. Figure 5-1 presents the schematic of spills within the FBC facility. The spills occurring during Phase I will be discussed further in a later section with the analytical results.

5.3 OBSERVED HEALTH AND SAFETY DETAILS

The health and safety protocol was designed to isolate the potentially toxic materials in the FBC process, protect personnel from possible contamination, and prevent the spread of suspected contaminated material. The observations made during Phase I indicate various aspects of personnel protection that require considerations for improvements to the Health and Safety Plan prior to initiation of Phase II of the FBC Project. In general, observed problems with adherence to the safety protocols were caused by an apparent lack of attention to approved procedures by some project personnel, inadequate facility accommodations and personnel preparation because of project scheduling, and facility limitations within the EPA G High Bay Area.

5.3.1 Test Day, Thursday, May 5, 1983 Observations

Observations made by health and safety observers during test day, Thursday, May 5, 1983 produced the following recommendations for project personnel:

- use splashguards, splash goggles, or face shields during mixing operation in the fuel feed shed,
- locate a protective equipment disposal container adjacent to the fuel feed shed during mixing operations,
- establish a specific location for storage of street and safety shoes in a locker other than the safety supply cabinet,
- post flammable liquid and/or no smoking sign at fuel feed shed,

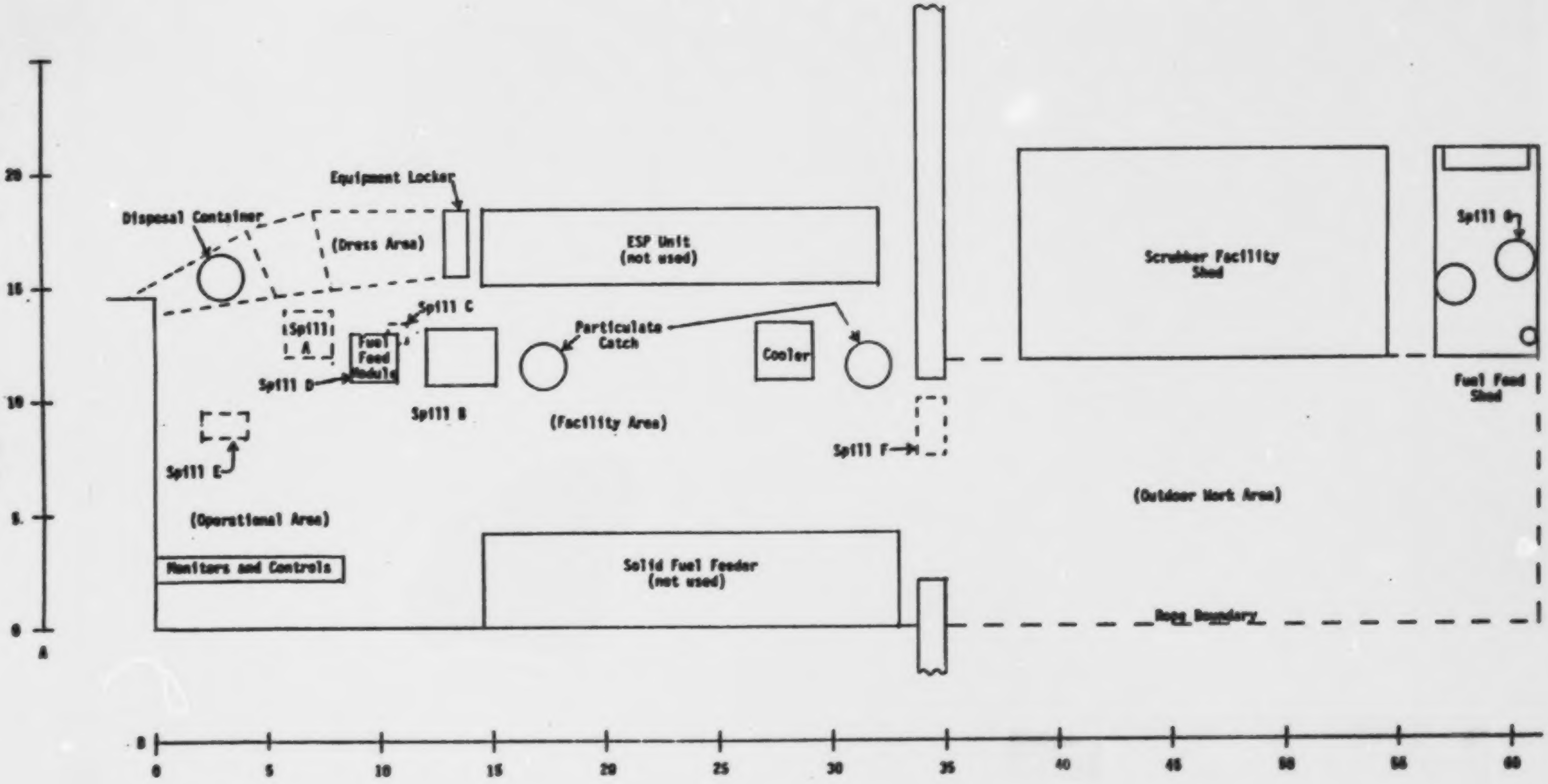

Equipment Locker
Disposal Container
(Dress Area)
ESP Unit
(not used)
Scrubber Facility
Shed
Spill G
Spill C
Spill A
Fuel
Feed
Module
Spill D
Particulate
Catch
Cooler
Spill B
(Facility Area)
Fuel Feed
Shed
Spill F
Spill E
(Operational Area)
(Outdoor Work Area)
Monitors and Controls
Solid Fuel Feeder
(not used)
Rope Boundary
20
15
10
5
0
A
B
0
5
10
15
20
25
30
35
40
45
50
55
60

Figure 5-1. Spill and wipe sample location schematic (5/25/83).

- establish a log documenting estimated exposure time in fuel feed shed,
- limit the involvement of third (clean) person during fuel mixing, and
- hardhats should not be required unless work is being done overhead on combustor superstructure.

The above recommended items pertained to personnel activities and to facility accommodations at the fuel feed shed with the exception of the hardhat requirement. The original protocol requiring hardhats for project personnel working on the ground floor at the FBC facility was perceived as nonessential, because, the overhead work in the mezzanine area is not directly above or accessible to the facility operators. Also, decontamination procedures would be required if the hardhats used were not disposable. All the recommended items were discussed with the appropriate project participants. Placard caution notices were posted at the fuel feed shed. "No Smoking/Flammable Liquid" signs were posted on the two exposed sides and on the center of the door. The log of personnel exposure time in the fuel feed shed was documented during the observation by the presiding safety observer. Table 5-1 provides a summary of exposure time and compounds. An interpretation of the protocol concerning the limited involvement of the third (clean) person during mixing activities was resolved. The involvement of the clean person would be to bring supplies or equipment if needed by the mixing team and would not include active assistance within the fuel shed.

All of the above recommended items for improvements received attention with the exception of the splash goggles or face shield. This item was accomplished after test day May 10, 1983.

TABLE 5-1. PERSONNEL EXPOSURE TIME IN THE FUEL FEED SHED

Test day	5/05/83		5/10/82	5/12/83	5/17/82	
Test period	1		1	1	2	
	Mixing	Sampling	Sampling	Sampling	Mixing	Sampling
Minutes exposed:						
Rudi Machilek/Acurex	35	5	9	9	11	6
Russ Clayton/Acurex	*	0	9	9	*	0
Ray Merrill/TSS	35	0	0	0	11	0
Herb Dempsey/Acurex	*					

*Clean person during mixing operation.

Test Period 1 - Chemicals exposed to: nitrotoluene, triphenylmethane, triphenoxy-s-triazine, isophorene.

Test Period 2 - Chemicals exposed to: tetrahydrofuran, methyl vinyl ketone, residue levels from test period 1 chemicals.

5.3.2 Test Day, Tuesday, May 10, 1983 Observations

Observation made by health and safety observers and discussion with project personnel through test day, Tuesday, May 10, 1983 produced the following recommendations:

- use face shields or splash goggles during sampling operations in the fuel feed shed,
- designate a clean/contaminated system for the miscellaneous tools and hardware utilized around the operational area,
- utilize lined containers for all contaminated glassware and trash, especially in the fuel feed shed and sampling area,
- install eye wash facilities,
- consider a provision for physical isolation of the FBC facility,
- designate with physical markings a dress area and a clean/contaminated shoe area,
- reemphasize the dress/undress procedure - describing the area and sequence of clean/contaminated dress guidelines,
- properly store the respirators used by the fuel samplers in a clean and sanitary fashion between sampling intervals,
- arrange available respirator and disposable gloves for the sampling teams at their sampling sites,
- properly wear protective clothing during recovery of sampling trains (coveralls should be worn as designed),
- mark spill areas as contaminated until analysis proves different (a record of spills sites should be maintained with a spill log completed),
- analyze hardware removed from FBC process (e.g. clogged probe, totalizer, and Y strain) for contamination before storing,
- analyze recycled fuel oil "solvent" barrel used for clean-up procedures,
- plumb SASS train cooling water to a drain,
- wear gloves, glasses, and safety shoes during recovery of SASS train sample,

- wear respirator during probe removal of sampling trains, and
- inform the EPA extra personnel (i.e. clean gopher) of his added congestion during the recovery operation and inadequate dress for the restricted area.

The recommended items were discussed with the pertinent project participants for action or protocol interpretation. The Acurex Safety Officer took appropriate action for the items related to facility operations. Goggles are now required for personnel involved in "waste" mixing and sampling in the fuel feed shed. Contaminated tools are cleaned in a designated solvent tank, rinsed with cleaned solvent, and dried before being returned to the tool box. Random wipe samples of the tools were taken to verify efficiency of the decontamination procedures. The wipe sample results are presented in Section 5.4. Lined containers have been provided as disposable receptacles in the fuel feed shed. The operational level eye wash station was installed and the mezzanine level eye wash station is available but had not been installed through the second test day. A dress area was designated with a dress procedure established for donning and doffing safety outfits. The facility operator devised a system for storing the disposal respirators during non-use periods so that it could be used through a test day.

The original protocols were supplemented with the addition of a spill chart and log for maintaining information of contaminated areas. The wipe sampling of potentially contaminated facility hardware and tools was also supplemented for verification of the decontamination efficiency.

Recommended items pertaining to the source sampling activities concentrated upon personal attention to details on the part of sampling personnel to safety protocols. The proper wearing of protective clothing is an essential detail in the safety program. The original protocol consideration of wearing gloves, respirators, and glasses during activities with potentially contaminated sampling parts or open ports is considered necessary. The overlooked items of the SASS cooling water drain and the EPA clean personnel were adequately resolved through project supervision informing the involved sampling personnel.

Recommended improvement of the listed items which require some further attention were as follows. The sampling team members were not observing the details of the clothing program. Sampling personnel were not wearing gloves during the handling of potentially contaminated material. Respirators were not accessible to sampling personnel while the sampling ports were open and waste was being combusted. A system for transporting contaminated sampling items and samples from the sampling and fuel feed area to the laboratory for analysis or cleaning needed development and further attention.

5.3.3 Test Day, Thursday, May 12, 1983 Observations

Observations made by health and safety personnel and discussion with project personnel through test day, Thursday, May 12, 1983 displayed many of the recommendations achieved or in the process of completion. These modifications are discussed with test day, May 17, 1983 in the following subsection. A major point observed on May 12 was the operation of the FBC incinerator at extreme high temperatures. The operation of the incinerator with a high feed rate to burn off excess spiked fuel caused high temperature which exposed the operators and facility to potential hazardous conditions.

5.3.4 Test Day, Tuesday, May 17, 1983 Observations

Observation and discussion with project personnel through the final test day of Phase I, Tuesday, May 17, 1983 produced the following recommendations:

- consider a provision for physical isolation of the FBC facility,
- install eye wash facility in sampling area,
- specify to each sample team member when the gloves or respirators are required and how to store when not being used,
- equip the fuel feed shed door with an apparatus for securing in an open position,
- provide an adequate supply of the heavier type gloves for activities which require handling of chemicals, chemical laden fuel, and samples,
- use shoe covers or dedicated safety shoes at the sampling team site,
- provide a system for containing contaminated items such as exposed sample train parts, chemical bottles, and fuel sample bottles for transporting to lab facilities,
- consider an improved means for controlling the scrubber facility activities; scrubber personnel and process upsets interfere with the integrity of the area,
- consider a facility modification for fabricating a pathway directly between the operations and sampling areas,
- consider equipping the operations and sampling areas with a direct line of communication to each other and outside locations,
- vent VOST train exhaust to the stack,

- dispose of potentially contaminated items at sampling sites (e.g., gloves, rags, and insulation material),
- remove idle sampling equipment from the area for alleviating congestion and contamination problems,
- include in sampling methodology the equipment necessary for capping the probes and handling contaminated glassware,
- determine the merit of the established safety protocols during probe insertion and removal. The value of wearing the respirator, gloves, and glasses during activities with the port cover removed need to be assessed.

The recommended items for the final test day of Phase I have been discussed with pertinent project personnel. The aspects noted concerning personnel details will require further attention in order to comply with prescribed and accepted safety protocols. The established safety protocols should not be ignored, but discussed at all project levels if situations seem inappropriate. The obvious facility limitations of the FBC process within the EPA G High Bay Area will require considerations for further project phases.

5.3.5 Observation Made After Test Period

Clean-up and maintenance activities were required at the FBC facility after completion of the last test day (May 17, 1983) of the Phase I test period. Wipe samples and analysis were performed for checking potential contaminated areas after clean-up procedures were completed. Analysis results determined the integrity of the FBC facility and the efficiency of clean-up procedures.

Wednesday, May 18, 1983 was utilized for facility maintenance. Two facility personnel were on-site but no clean-up or open system activities were performed. Therefore, safety clothing and procedures were not required.

The spill areas were marked off and labeled for potential contamination on Friday, May 19, 1983. These marked areas were designated for decontamination purposes. A quantitized contamination level was determined for each area. These decontamination activities were completed after completion of the facility maintenance activities.

The material samples from the FBC process were obtained on Friday, May 20, 1983. Samples were taken on the bed material and particulate material from the two cyclones. The sampling required three personnel to dress in safety clothing and follow safety procedures while obtaining samples from the open system. The analysis of the samples were negative, therefore, the maintenance operation to remove the bed material and cyclone particulate catch did not require implementation of full scale safety procedures.

Final post-operational Phase I facility decontamination samples were on May 26 by TRW to verify the absence of contamination from several areas. A total of 12 filter-wipe samples were collected and analyzed (TRW correspondence T310.JFM.83.009). The positive analytical results (discussed in Section 5.4) from 3 samples taken within the fuel feed shed resulted in a recleaning of the shed by facility personnel in an attempt to completely decontaminate the area. On June 16 two additional wipe samples were collected verifying the adequacy of the additional clean-up activity.

5.4 ANALYTICAL RESULTS OF ASSOCIATED HEALTH AND SAFETY SAMPLES DURING PHASE I

Figure 5-1 shows locations of process spills and contamination checks. A spill log was maintained for documenting location, occurrence, clean-up procedure, personnel involvement, and analytical results of the wipe samples performed after decontamination of the area. A record of the exposure time in the fuel shed was maintained in the TRW health and safety notebook. Table 5-2 is the revised spill log indicating the results of wipe-samples at the designated spill areas. The analyses performed by TSS and TRW are discussed in the appended wipe sample notes (correspondence R. Merrill, 5/19/83) contained in Appendix D.

The positive results of the spill areas in the May 5 and 10 wipe samples indicated the necessity in developing a more effective clean-up procedure. The procedure was investigated by the project analytical and safety monitors. The spill areas with positive results were the controls for testing the developed procedures. The analytical results of the modified clean-up procedures are presented in Table 5-2 and the modified and approved clean-up procedure for the remaining project phases is included in Appendix C.

Contamination checks of facility items that had potential exposure during project activities were performed. The items checked were tools used for facility maintenance, FBC incinerator parts removed from the process (after first test burn), and the receptacle for disposable material. The tools and parts had been cleaned with solvent (fuel oil). But, the solvent had not dried on the parts, therefore, causing analytical difficulties. The two wipe samples of the tools and receptacle were negative when screened by gas chromatography flame ionization at 15 nanogram (ng) detection limit. The analytical procedures followed by TRW are specified in the appropriate correspondence (see Appendix D - TRW T310.JFM.83.007).

TABLE 5-2. REVISED SPILL LOG FBC PHASE I TESTING (6/24/83)

Spill	Location	A/B Coordinates	Date	Time	Clean-Up Personnel/Procedure	Designated Spill Area Dimensions	Contaminated Status
A	Floor below coolant regulator	14/7	5/05/83	1930	R. Merrill - Sawdust and methylene chloride	24" x 24"	Yes
AA	Repeated clean-up after contaminated analysis	14/7	5/06/83	0800	R. Clayton and R. Machilek - Fuel oil and isopropyl (IPA)	24" x 24"	Yes
B	Floor in pathway (≈ 3 ft) east of FBC unit	9/12	5/06/83	0930	R. Clayton - Fuel oil and IPA	b	No
C	Floor below NE corner of fuel feed module	13/11	5/10/83	1530	D. Sanchez - Fuel oil and acetone	12" x 12"	Yes
D	Inside fuel feed module	12/9	5/10/83	1700	R. Clayton - Fuel oil and IPA	24" x 24"	Yes
E	Floor below and beside process analyzers	9/3	5/17/83	1100	R. Mackilek - Saw dust	12" x 24"	No
F	Bay door	9/33	c		None	24" x 12"	No
AAA	Repeated clean-up after contaminated analysis	14/7	5/26/83	d	H. Dempsey - Revised procedure (Appendix C)	24" x 24"	No
CC	Repeated clean-up after contaminated analysis	14/7	5/26/83	d	H. Dempsey - Revised procedure (Appendix C)	12" x 12"	Yes
G	Fuel feed tank top	16/62	5/26/83	d	None	e	Yes
GG	Repeated clean-up after contaminated analysis	16/62	6/16/83	d	H. Dempsey - Revised procedure (Appendix C)	e	Yes

[a] Coordinates from facility schematic.

[b] Three or four drops not dimensioned.

[c] Tools placed here after decontamination.

[d] Repeated cleanup.

[e] Circular spill area of 2.76 ft^2.

Analysis of personnel monitor badges were performed under subcontract to Northrop Services, Inc., Research Triangle Park, North Carolina. All personnel monitors produced negative results for five of the investigated compounds. Table 5-3 provides the minimum detection of the analyses. Appendix D present the Northrop Analytical Chemistry Laboratory Test Results. High background levels were evident during the analysis procedure with the passive dosimeters utilized for personnel monitors. The dosimeters maintained as a blank had the same problem with high background levels. Therefore, a new system for personnel monitoring is suggested with active dosimeters replacing the passive dosimeters.

The area monitors were utilized only during the volatile compound burn of Phase I (May 17, 1983). The monitors were operated in the fuel feed shed and on the mezzanine in the source sampling area. The results of the analysis on the two area samples collected during the volatile testing are presented in Table 5-4. A design feature deficient in the area monitor system was the capability for measuring the accurate volume of sample. The sample pump and flow measuring device needs to be at the same functional volumetric range.

A potential problem with the area monitor sampling was the possibility of influences from other sources in the G High Bay area besides the FBC process. These additional activities may heighten the background levels and may interfere with the integrity of the area monitor sample.

Contamination checks were performed at various locations within the FBC facility for potential spills and exposure. The locations chosen were areas exposed to open process streams and feed stock chemicals.

TABLE 5-3. NORTHROP MINIMUM DETECTION LIMITS OF PERSONAL MONITOR ANALYSIS

Chemical	Detection Level
4-Nitrotoluene	20 ng
Isophorone	10 ng
α,α,α,p-Tetrachlorotoluene	10 ng
Methyl Vinyl Ketone	10 ng
Tetrahydrofuran	10 ng
Triphenylmethane	No standard obtained
2,4,6-Triphenoxy-s-triazine	No standard obtained

TABLE 5-4. FBC PHASE I: AMBIENT AREA MONITOR ANALYTICAL RESULTS (TSS)

	VOST Results from Ambient Air Samples (mg per cubic meter)	
Area	Methyl Vinyl Ketone	Tetrahydrofuran
Sampling Bay	None Found	0.05
Fuel Shed	0.04	0.15
Blanks	ND*	ND*

*Tenax-Carbon cartridges were used and no material was detected; however, unidentified levels of unquantified background compounds were encountered.

The top of the feed barrel location was designated a spill area (G on spill table and chart) after analysis showed high contamination levels occurred during mixing operations.

TRW collected the contamination samples on May 26 and repeated sampling after decontamination procedures on spill location G on June 16. The sampling locations are described in Appendix D (TRW Memo T310.JFM.009). The positive results were reported for wipe samples obtained from the recycle line, from Spill Area C, from the top of the clean fuel drum, and from the fuel feed drum (Spill G).

The June 16 sampling included a check on the inside of the fuel feed shed. A wipe sample of the inside shed walls was negative at the 15 nanogram detection level.

Two sites within the FBC facility still have positive results. The top of the feed barrel (Spill G) and the spill area below the feed module (Spill C) still show a detectable, although reduced, amount of triphenylmethane after several decontamination attempts. The significance of the residual triphenylmethane levels in these two areas has not been determined at this time.

5.5 HEALTH AND SAFETY PROGRAM COST ANALYSIS

The maintenance and operation of the FBC test facilities in an exemplary manner is an additional cost to the overall project. This section will provide an estimation of the cost for implementing and observing the health and safety procedures during Phase I activities. The cost of safety-related equipment and estimated manhours for safety-related personnel will be provided.

Table 5-5 lists the safety equipment used during the Phase I test period from May 5 to May 24, 1983. Table 5-6 sums the total cost for Phase I safety equipment with breakdowns of the cost per test day and per maintenance day. The calculated average equipment cost was $17.38 per person during a test day and $16.15 per person during a maintenance day (i.e., days with potential contamination from the process). No extensive amount of equipment was used after the negative process material results were processed.

The manpower costs for personnel maintaining and observing the safety procedures can be categorized into four roles. These roles can be considered as an additional workload greater than that required of normal project activities. These safety related roles are a Health and Safety Coordinator, Health and Safety Observers, Contamination Samplers and Analysts, and the required clean personnel standing by to assist project personnel in the event of a spill or potential contamination event.

Acurex provided the Health and Safety Coordinator. This person also fulfilled additional roles as an observer and a clean person during the Phase I test period. Therefore, considering that the coordinator fulfilled three roles, the combined personnel cost would be as a full time project participant. Approximately 80 manhours were required during the testing period of May 5 to May 18.

The use of two independent observers were utilized for monitoring the project health and safety procedures. TRW provided one observer and the Acurex coordinator was the other. Both observers completed two or three checklist observation per test day and were present during cleanup

TABLE 5-5. PHASE I HEALTH AND SAFETY EQUIPMENT

	5/5	5/6	5/9	5/10	5/11	5/12	5/13	5/16	5/17	5/18	5/19	5/20	5/23	5/24
No. of personnel	9	2	3	9	2	9	2	3	6	2	2	3	2	2
Respirators	6	2	3	4		4	2	3	2		2	3		
Gloves (neoprene)	2		1	2		5	2	2	5		2	2	2	2
Gloves	26	2	2	26		20		1	20			1		
Overalls	21	2	3	19		19	2	3	15		2	3		
Shoe covers	15	2	3	13		13	2	3	10		2	3		
Head covers	21	2	3	19		19	2	3	15		2	3		
Goggles[a]						2								
Test activity (M/NM)[b]	X M			X NM		X			X M					
Non-test activity (MT/C)[c]		X C	X MT/C		MT		C	X MT		X MT	X C	X MT/C	X MT	X C[d]

[a] Cleaned and recycled.

[b] Mixing or non-mixing.

[c] Maintenance/clean-up.

[d] Non-contaminated materials.

TABLE 5-6. SAFETY AND HEALTH EQUIPMENT COSTS FOR PHASE I TEST PERIOD

	Cost/ Item	Total Used	Cost/Phase I Test Period	Average Test Day	Cost/ Test Day	Average Clean-up and Maintenance Day	Cost/Clean-up Day and Maintenance Day
Respirators	$10.00	30	$300.00	4	$ 40.00	3	$30.00
Gloves (neoprene)	2.50	27	67.50	4	10.00	2	5.00
Gloves	.13	97	12.61	23	2.99	1	.13
Overalls	3.66	89	325.74	19	69.54	3	10.98
Shoe covers	.44	66	29.04	13	5.72	3	1.32
Head covers	.34	89	30.26	14	4.76	3	1.02
Goggles	3.00	2	6.00	2	6.00	0	0
TOTAL COST			$771.15		$139.01[a]		$48.45[b]

[a] Assuming eight project personnel at the FBC facility during an average test day, the average cost per person for safety equipment was $17.38.

[b] Assuming three project personnel at the FBC facility during an average clean-up and maintenance day, the average cost per person for safety equipment was $16.15.

activities. The checklist observations and documentation required approximately six hours per test day and four hours per clean-up activity. Since the Acurex observer was also the coordinator, the additional cost to the project was only the TRW observers. Approximately 167 manhours for on-site observations and safety reporting and documentations were expended by TRW.

Safety related sampling analysis were performed by EPA/TSS, TRW, and Northrop Services. EPA/TSS and TRW performed wipesamples and material sampling with the analysis. Safety related EPA/TSS activities involved approximately 40 manhours. The monitoring badge analysis by Northrop expended approximately 100 manhours. The sampling and analysis by TRW laboratory personnel expended approximately 44 manhours.

The requirements of a clean person during fuel mixing/sampling and source sampling operation provided an additional cost to the project. The clean person at the feed shed was either a facility operator or the Health and Safety Coordinator. Therefore, no additional personnel was required. The clean person at the source sampling area was utilized on test days. This was an additional person that should have been accessible full-time for the assisting contaminated sampling personnel. Therefore, 80 manhours should be attributed to the project for this function.

A total manhour expenditure for implementing and maintaining the Phase I health and safety plan was 511 manhours. This is an estimate based on the aforemention health and safety roles.

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 FACILITY

The fluidized bed combustor was found to provide a safe and efficient means of destroying hazardous wastes. As with any incineration process the effect on formation of products of incomplete combustion (PICs) is unknown. Further studies are recommended to provide in-depth empirical data on the effects of various operating parameters on the destruction of surrogate wastes. These studies should include facility characterization and optimization testing to evaluate the parametric effects on operation. The first task of Phase II of this program will be a thorough review and evaluation effort to determine the direction and goals of future testing.

Findings from Phase I testing have shown some deficiencies in the facility which should be addressed and/or corrected during Phase II. These include:

- Difficulty in cleaning or changing fuel feed nozzle without dumping bed material. On two occasions during the test series, it was necessary to clean the fuel feed nozzle. In one case, the nozzle with its cooling jacket had to be removed from the bed during a test series when it was desirable to retain the bed material in the combustor. The probe was removed, repaired and reinstalled while losing only 3 liters of bed material. This procedure is workable but a system designed for ease of maintenance would be preferable and would eliminate the necessity to handle potentially hazardous material.

- The pressure drop through the bed decreased for each test, indicating a loss of bed material. One possibility is that a quantity of bed material fell through the distributor plate holes following each shut-down. The .100-inch holes allow a small quantity of the .035-inch spheres to fall through before bridging the gap. The material loss could be aggravated by vibration of the facility which occurs during normal operation or when maintenance is being performed. Studies with differing material sizes or distributor designs could result in a minimization of the problem. An alternative solution is to redesign the plenum to recover bed material and recycle it along with the cyclone catch.

- Increased air supply for cold startup is a critical improvement to be incorporated prior to Phase II testing. The blower was required to operate at an overcurrent condition for two hours during each start-up. The dense bed material requires a large volume of air to initiate fluidization. The repair of leaks in the electric heaters may be an adequate solution to the problem if it is determined that leaks will not reappear. An alternative fix would be the replacement of the blower motor with a higher horsepower version. Another possibility is the introduction of compressed process air during the initial start-up period to facilitate fluidization.

- The fuel/waste mixing procedure could be improved by an injection system engineered to eliminate the handling of open containers of test compounds in the facility area. Such a system would be particularly useful in mixing volatile compounds. A sealed, valved container could be filled in a flume hood, transported to the fuel shed and installed in the feed loop so that mixing would occur when the pump was operated.

- A bypass loop around the inlet air orifice would be beneficial during start-up. The fluidization air requirements for a cold bed are much greater than when the unit is at operating temperature. In order to accurately measure the flow rate into the combustor during operation, a small orifice is required. During start-up however, the small orifice produces a large pressure drop which has to be overcome by an overtaxed blower.

- Data collection could be improved by the replacement of the multipoint temperature recorder with a newer, more maintenance-free model and the addition of an automatic recorder for fuel flow rate or an accurate flow totalizer. The latter item was planned for Phase I but not deemed essential to the test.

- The addition of several pilot lights for facility control would be advantageous to quick determinations of operational conditions. Lights are desirable for each safety interlock on the gas and oil feed systems, oil feed pumps and oil feed shut-off valves. An audible alarm should be installed on the oil feed shut-off valve.

- As determined by the difficulty in cleaning the floor in the facility area after spills, an impervious sealant should be applied to the concrete floor. The likely choices for sealants are epoxy paint or polyurethane coatings. Similarly, the wood floor in the fuel feed shed should be sealed. The existing floor drains should be filled and replaced by sealable drains to prevent any spills from contaminating the building drain system.

- More storage area is needed in the facility area to avoid the difficulties of needing materials outside of the facility area while dressed in protective clothing. Similarly, personnel access between the facility area and the mezzanine sampling area is necessary to prevent the traffic and possible contamination of areas outside of the designated hazardous areas. Solutions to these problems could be achieved by the removal of the large ESP unit adjacent to the facility. A barrier wall could be built and lined with storage cabinets. Space would then be available to place a stairway to the mezzanine along the outside wall beside the baghouse. Other possibilities will be explored. These problems will be further discussed in the Health and Safety report.

- The installation of a telephone in the facility control area is recommended to improve communication with all parties involved in the test program. A request has been submitted to building management for the telephone. An intercom would be beneficial to aid the communication between the facility operators and sampling crew.

- The installation of sample ports downstream of the flue gas cooler or an alternate means of cooling may be necessary to allow the operation of the unit at higher combustor temperatures. The requirements will be coordinated with the Technical Support Staff to determine what, if any, modifications are necessary.

- As proposed for Phase II, the installation of an area exhaust fan and appropriate physical separations from the other facilities in the High Bay building are advised. This action should be taken to avoid any fugitive emissions caused by spills or facility upset conditions escaping the area.

- The plexiglass enclosures for the fuel pumping and control system should be resealed with materials compatible with fuel oils and solvents used for clean up.

- Insulation of the baghouse and/or exhaust stack is necessary to maintain exit temperatures above 100°C to prevent condensation in the stack. This change is of particular importance if chlorinated compounds are burned without a scrubber to remove the HCl. Attention must also be given to the cooling tube bundles in the flue gas cooler under such conditions since the tube surfaces will also be below condensation temperatures and subject to corrosion.

- Facility maintenance would be improved by the acquisition of selected tools necessary for the fuel feed system which would be dedicated for use on potentially contaminated components. These tools would be maintained in the fuel feed shed to prevent contamination of "clean" components.

- A waste oil container and storage location should be established to receive clean up solvent to eventually be mixed with fuel oil and burned in the FBC.

These problem areas will be addressed during the Phase I evaluation period and Phase II planning.

6.2 SAMPLING AND ANALYSIS

The successful completion of the sampling and analysis portions of the test series led to the following conclusions:

- Sorbent sampling through methods such as SASS was proven to be an effective means of capturing semi-volatile and condensible organics.

- The Glass SASS was effective in HCl sample capture with no degradation in train components.

- Analysis of SASS samples should be completed within three weeks of the sampling to minimize any laboratory contamination.

- Dilution tunnel (SDSS) sampling is effective, as indicated in laboratory studies, in the collection of particulate-bound organics but was not effective in semi-volatile organics capture.

- Three replicates were found to be adequate to meet QA requirements for VOST samples.
- VOST samples can be frozen for at least one week without affecting the analysis.
- VOST cartridges should be cleaned and blank checked immediately prior to use.
- Carbon-Tenax cartridges should be divided, keeping carbon and Tenax separate to avoid interference in GC/MS analysis.
- VOST glassware present problems with its fragility. More rugged construction would aid in workability.
- Inlet or fuel samples should be taken and labelled to coincide with the VOST sampling.

These findings will be incorporated in the planning for future testing.

6.3 PROPOSED HEALTH AND SAFETY RECOMMENDATIONS AND REFINEMENTS

Recommendations for improving the health and safety program may require modifications of original health and safety protocols and physical changes of the FBC facility. The recommendations presented here are based on TRW and Acurex Safety Monitors observations during facility operations, maintenance, and checklist reviews of Phase I activities and discussion with pertinent project personnel.

Some observed variances from the originally planned safety protocols were questioned for feasibility. These variances were discussed with pertinent project personnel for consideration in modifying the procedures. The wearing of respirators and disposable gloves during sampling activities with the sampling port open was considered. It was suggested that the negative pressure of the source would deem the respirators unnecessary. However, slight pressure fluctuations due to process upsets are possible. Therefore, a respirator should be available for use or be used for sampling personnel at open port sites. The disposing of this respirator for each 8 hour period in this application, should not be required if stored properly. Un-opened respirator packages need no special storage accommodations. An opened respirator recycled after less than 8 hours use requires storage in a sealed container to prevent possible contamination.

The disposable gloves are not appropriate for handing sampling equipment at their high temperatures of operations but heat resistant (asbestos) gloves when used, should be handled as if contaminated. The use of asbestos gloves are not permitted under the current plan, unless the gloves are diposed of after each use.

The question of disposing of the safety suit each time the personnel leaves the site was considered. The recycling of safety suits is not permitted because of potential contamination. The frequent trips from the area could be alleviated with sufficient preparation, planning, and utilization of the clean person. A direct communication line in the FBC areas (operational and sampling) could provide assistance from clean personnel if preparation and planning are not sufficient to minimize traffic to and from the restricted area.

Facility additions and modifications are necessary for improving safety protocols. The dress area could be modified with an entrance for clean personnel and a separate exit for contaminated personnel to doff equipment and depart. Recommended facility additions include a permanent eyewash station on the ground floor and wash-up facilities within the restricted FBC area. The installation of a permanent eyewash will ensure availability at all times of a fresh hygienic wash solution. The installation of a wash-up area in G-219 is recommended with both lavoratories and showers which will eliminate the need to leave the FBC area prior to decontamination. In order to facilitate a rapid and complete clean-up of minor spills with the operational area, the present concrete floor should be sealed. This procedure may eliminate the sorption of compounds into the concrete and allow a complete decontamination of a spill area.

The installation of an activated carbon protected positive ventilation system is a high priority for installation at the fuel feed shed. The interaction with the building system has the potential of becoming critical during the mixing and testing of volatile compounds. Results of area monitors and shed wipe samples will determine the background levels.

In addition, it is recommended that a provision be made to ventilate and to maintain the integrity of the FBC facility air flow. This would require modifications to the EPA G High Bay Area. The ventilation of the FBC area with air flow from the high bay area to an exhaust at the facility should proceed as planned. The integrity and effectiveness of the safety ventilation modifications are questionable when the roll-up door is open (i.e. a wind current from the north can create conditions where air flow is from the outside through the FBC into the high bay area). Installation of a standard door would minimize this effect and still allow the necessary access to the fuel feed shed. In addition this modification would allow winter operation of the facility.

The congestion in the sampling area (mezzanine level) causes persistent health and safety problems. To a degree crowding in this area can be improved by removing certain process and sampling equipment from the site. Possible changes include:

1. temporary or permanent removal of the ESP power packs,
2. relocation of scrubber belt filter to the ground level, and
3. relocation of scrubber stack gas analyzer to ground floor.

Coordination within EPA is required and possible site (for item 2 and 3 above) is the present location of the wind tunnel facility. This site will be available as the wind tunnel facility is removed for installation of a spray dryer.

Egress paths and potential contaminations problems could be reduced by:

1. installing a permanent electric service to the source sampling area to eliminate the need for the maze of temporary cords,
2. fabricate the SASS set-up with the metering module outside the sampling area, and
3. removing sampling equipment from the area when not part of test phase.

An alternate proposal is the fabrication of another modularized sampling area. This sampling area could be constructed at the mezzanine level but on the opposite side of the current site. The activity in the scrubber area would be insulated from the FBC activity and physical boundaries could be maintained. A direct path between the two levels could be included thereby integrating the facility into a complete unit.

Possible modifications to the FBC incinerator would provide security in operating the unit and in performing maintenance if required. The provisions include:

1. the waste injection nozzle should be redesigned to allow removal for cleaning/repair independent of facility operational status (Plenum redesign/fabrication may be required),
2. provision for fully sparing or rapid replacement of feed nozzle should be considered in redesign, and
3. consideration should be given to the addition of a "double wall" arrangement in the positive pressure area of the combustor to facilitate collection and venting of possible fugitive emissions from the positive pressure regions of the combustor.

A minor modification is the addition of an apparatus at the fuel feed shed for securing the door in an open position while personnel are inside sampling or mixing the fuel. The requested foot operated disposal receptable is essential for the immediate and unhampered removal of contaminated gloves after fuel handling activities. A more appropriate supply of the heavier quality gloves is also necessary for personnel mixing and handling fuel samples.

Shoe covers should be reconsidered with more durable disposable cover-ups chosen. The disposable covers would be utilized instead of or in addition to the "dedicated" safety shoes system that is not presently effective.

The sampling system used for area monitors should be upgraded with associated gas sampling equipment dedicated to the facility. A prescribed container for carrying sample and potential contaminated equipment should be designed and maintained for transportation of fuel and source samples to and from the facility.

The refinements and recommendations presented will improve the integrity of the FBC facility and the success of the health and safety guidelines. The modification for further isolating the FBC facility will provide control of the area (people and physical aspects) and physical boundaries for the proper application of safety and health procedures. The imaginary boundary concept implemented during Phase I has not succeeded in controlling the traffic in and out of the area. The prevention of the scrubber personnel and FBC sampling crew movement throughout the area and through other parts of the building will require distinct physical boundaries.

All project personnel should be made aware of the need for a good health and safety plan and of the consequences to themselves and other of poor practice of the prescribed plan. The training program provided an example and the personnel should practice the details in accordance with proper personal hygiene practices. The effectiveness of the program will depend on the degree to which personnel accept the training, willingly put the principles to use, and cooperate in preventing exposures and the creator of potentially hazardous situations.

APPENDIX A

FBC PHASE I HEALTH AND SAFETY CHECKLIST

(EXAMPLE)

Approved 5/6/83

Date ____________

Time ____________

Observer ____________

FBC PHASE I HEALTH & SAFETY CHECKLIST

A. FUEL FEED SHED

1. Is the fuel shed ventilation and monitoring system being operated according to prescribed specifications?

   Rating: 1 2 3

   Comments: ____________

2. Are the personnel with access to the fuel feed shed donning protective clothing and respirators at all designated times?

   Rating: 1 2 3

   Comments: ____________

   What is the quality of protective clothing (suitability)? ____________

   What improvements are possible? ____________

3. Are spills handled (controlled and cleanup) promptly and in the correct manner?

   Rating: 1 2 3

   Comments: ____________

4. In the event of a spill, are decontamination protocols detailed enough to provide clear guidance?

   Rating: 1 2 3

   Comments: ____________

Date ______________

Time ______________

Observer ______________

FBC PHASE I HEALTH & SAFETY CHECKLIST

B. FACILITY OPERATION

1. Are the floors, aisles, and control equipment kept clear of unneeded miscellaneous articles?

   Rating: 1 2 3

   Comments: ____

   ____

   ____

2. Are unauthorized personnel excluded from the restricted area? Are visitors adequately restricted from entering the area?

   Rating: 1 2 3

   Comments: ____

   ____

   ____

3. Are contaminated tools handled per safety protocol in the advent of leakage from the unit:

   Rating: 1 2 3

   Comments: ____

   ____

   ____

4. Is the fire extinguisher and eye wash bottle located in an accessible location during facility operation?

   Rating: 1 2 3

   Comments: ____

   ____

   ____

Date _______________

Time _______________

Observer _______________

FBC PHASE I HEALTH & SAFETY CHECKLIST

C. SAMPLING ACTIVITIES

1. Is the space available adequate to safely operate the sampling trains?

   Rating: 1 2 3

   Comments: ____

2. Is the egress route sufficient to handle the equipment and personnel?

   Rating: 1 2 3

   Comments: ____

3. Are protective clothing worn during operation? What improvements are necessary?

   Rating: 1 2 3

   Comments: ____

4. Are respirators and suitable gloves worn during those times when the sampling trains are inserted or removed from the duct work during waste firing?

   Rating: 1 2 3

   Comments: ____

5. What are the environmental conditions?

   - Temperature __________
   - Humidity __________

   Rating: 1 2 3

   Comments: ____

C. SAMPLING ACTIVITIES (continued)

6. Are potentially contaminated equipment handled in a suitable manner?

Rating: 1 2 3

Comments: ______________________________

Date ______________

Time ______________

Observer ______________

FBC PHASE I HEALTH & SAFETY CHECKLIST

D. OVERALL

1. Are cleanup areas suitable?

Rating: 1 2 3

Comments: ______________________________

2. Are emergency self-contained breathing apparatus located in their prescribed position?

Rating: 1 2 3

Comments: ______________________________

3. Are emergency numbers and warning signs posted and a Roster maintained on the bulletin board during operation?

Rating: 1 2 3

Comments: ______________________________

4. What is the overall adequacy of the facility?

Rating: 1 2 3

Comments: ______________________________

APPENDIX B

PROCEDURE FOR DONNING/DOFFING PERSONNEL SAFETY EQUIPMENT

PROCEDURES FOR DONNING/DOFFING PERSONNEL SAFETY EQUIPMENT

Donning:

1. Safety equipment should be donned prior to entering the FBC restricted areas.
2. The area immediately in front of the safety equipment locker has been designated as a clean area and should be utilized to don safety equipment (including shoe covers but not "contaminated" shoes).
3. Order of dress is not important EXCEPT "contaminated" shoes must be donned after all other equipment is in place.
4. "Contaminated" shoes can be donned without violating the restricted areas by stepping out of "clean" shoes in the clean area into "contaminated" shoes in the restricted area.

Doffing:

1. Safety equipment must be removed prior to leaving the FBC restricted areas.
2. Safety equipment should be removed in the areas adjacent to one of the contaminated equipment receptacles.[1]
3. Order of equipment removal is important. Carefully remove equipment in the following sequence ensuring that each article of clothing is turned inside out. Transfer disposable equipment to the contaminated equipment receptacle immediately.
    - goggles[2]
    - gloves[3]
    - head cover
    - respirator
    - coveralls
    - shoe covers
4. Violation of clean areas should be avoided during shoe cover removal by stepping into a clean area (from the restricted area) as each cover is removed. Similarly, by stepping from "contaminated" shoes in a restricted area into "clean" shoes in a clean area the doffing of contaminated safety equipment can be completed without violating designated clean areas.

Notes:

1. Since gloves have been shown to require frequent changing, a convenient receptacle has been provided near the waste feed shed.
2. Goggles should be thoroughly washed with soap and warm water.
3. Mixing and sampling operation in the fuel feed shed require gloves to be removed and disposed of first.

APPENDIX C

SPILL CLEAN UP PROCEDURE

ACUREX Corporation

Energy & Environmental Division

MEMORANDUM

TO: David Sanchez
EPA/IERL (MD-33)

FROM: Herb Dempsey

DATE: May 25, 1983

SUBJECT: FBC Phase I Spill Clean Up

During Phase I burn activities several accidental losses of potentially contaminated materials occurred. Analyses of wipe samples following clean up indicated that initial attempts to clean up the first "spill" (5/5/83) were unsuccessful (i.e. the wipe sample was positive). Several additional spills occurred and were cleaned up using a variety of techniques.

The positive wipe sample following spill clean up and the inconsistency of spill clean up techniques indicated a need to formulate a standardized clean up procedure. It was decided that once the procedure was developed, three of the contaminated areas would be recleaned and wipe samples taken to determine effectiveness of the clean up.

Ray Merrill and I defined the attached spill clean up procedure and spills A, C, and E were cleaned up today (5/25/83) per the newly established protocols. Apparatus utilized is shown in the attached sketch. The apparatus needs some additional optimization which should be addressed as part of the Phase II Health and Safety activities. Since the attached procedures only cover small spills, similar procedures should be developed to handle large spills.

cc: R. Clayton (Acurex)
B. Jongleux (TRW)
R. Merrill (EPA)
C. Stackhouse (TRW)

ACUREX CORPORATION: SOUTHEAST REGIONAL OFFICE 3200 NELSON-CHAPEL HILL HIGHWAY
P. O. BOX 13109 RESEARCH TRIANGLE PARK, NC 27709 PHONE 919-549-8915

HAZARDOUS SPILL CLEAN UP PROCEDURES

- Spills Less Than One Gallon -
- Flat Un-diked Surfaces -
5/25/83

1. Cover the spill immediately with sawdust or other sorbent to contain the spilled material.

2. Work the sorbent into the spill. Add more sorbent as required.

3. Pick up the contaminated sorbent and transfer to appropriate container (e.g. "poly bottle").

4. Repeat steps 1, 2 and 3 as required.

5. With appropriate solvent (water, propanol or acetone) clean a narrow area around the perimeter of the spill.

6. Construct a "dike" by applying a 4-6 mm bead of silicon rubber caulking around the spill area. Allow the dike to cure (several hours).

7. Cover the area within the dike with acetone. Allow the acetone to stand over the spill several minutes (5 to 10).

8. Using the apparatus described in attached diagram, pick up the contaminated acetone.

9. Repeat steps 7 and 8.

10. Direct a stream of acetone from a squeeze bottle over the spill area such that the flow of acetone washes the area in one direction to one corner of the diked area.

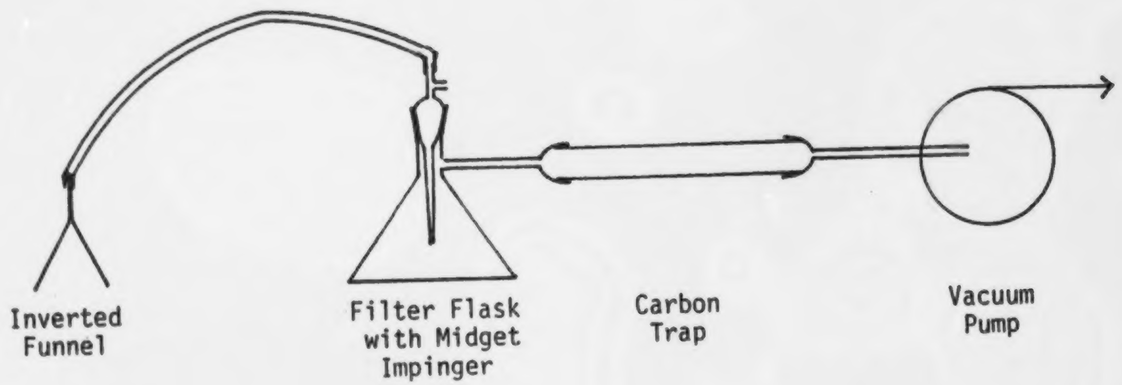
11. Concurrently, use the vacuum apparatus to pick up the wash stream.

12. Transfer contaminated acetone and other clean up material (e.g. Kimwipes, etc.) to appropriate containers (e.g. poly bottles).

13. Wash the clean up apparatus with at least three volumes of acetone (wash solution goes into poly bottle as waste).

14. Label waste containers and transfer to local incinerator for disposal.

15. Arrange for wipe sample and analysis.


Inverted Funnel
Filter Flask with Midget Impinger
Carbon Trap
Vacuum Pump

Small Spill Clean Up Apparatus

R. Merrill
5/19/83

WIPE SAMPLES NOTES

Two sets of wipe samples were received to be tested for the presence of known hazardous compounds (isophrone, chlorotoluene, nitrotoluene, and triphenylmethane) using gas chromatography. The first set was received on May 5, 1983 and consisted of samples B109D and B109E. The second set was received on May 10, 1983 and consisted of samples B112B, B112C, B112D, and B112E.*

The wipe samples as received consisted of a Whatman filter placed in a bottle. The filter was extracted by placing 10.0 ml of methylene chloride in the bottle and shaking vigorously for 2-3 minutes. The methylene chloride containing the filter extract was then placed in a 10 ml volumetric flask and brought to volume (some of the initial methylene chloride was absorbed by the filter paper). The samples were then injected into a gas chromatograph.

The resulting chromatograms were consulted in order to detect the possible presence of isophrone, chlorotoluene, nitrotoluene or triphenylmethane by comparison of retention times to a prepared standard containing those compounds. Since many of the wipe samples contained a great deal of organic material, the chromatograms were difficult to interpret. Therefore, triphenylmethane was chosen as a marker compound due to its relatively long retention time (13.56 min.) in comparison to many of the organics inherent in the wipe samples.

All of the wipe samples except B112B displayed a compound having a retention time equivalent to that of triphenylmethane.

---

*The spill number and location of the FBC facility schematic were added to the table of results.

REVISED WIPE SAMPLE RESULTS

Spill	Sample No.	Sampling Location	Location/ Coordinates	Possible Presence of Triphenylmethane
A	B109D	Wipe after cleanup under regulator	13.5/7	Yes
-	B109E	Fuel feed line rinse		Yes
B	B112B	Wipe in front of FBC	9/12	No
C	B112C	Wipe fuel, supply module	13/11	Yes
A	B112D	Wipe under valve	13.5/7	Yes
D	B112E	Wipe fuel feed box	12/10	Yes

NORTHROP SERVICES, INC. A Subsidiary of Northrop Corporation
ENVIRONMENTAL SCIENCES

P.O. Box 12313
Research Triangle Park, N. C. 27709
Telephone: (919) 549-0611

4240-83-2-1504

ANALYTICAL CHEMISTRY LABORATORY
Test Results

Date 6/15/83

Analysis minimum detection limit (MDL)

Sample Type Personal Monitors

Date Received various

For Jewell Finch
U.S.E.P.A.
MD-50
Research Triangle Park, NC 27711

	MDL
Nitrotoluene	20ng
Isophorone	10ng
Tetrachlorotoluene	10ng
Methyl Vinyl Ketone	10ng
Tetrahydrofuran	10ng
Triphenylmethane	No standard obtained
2,4,6-triphenoxy-s-triazine	No standard obtained

Remarks

Analyst Zora E. Bunn

RECEIVED MAY 23 1983

FBC-ΦI
Health & Safety

NORTHROP SERVICES, INC. A Subsidiary of Northrop Corporation

ENVIRONMENTAL SCIENCES

P.O. Box 12313
Research Triangle Park, N. C. 27709
Telephone: (919) 549-0611

4240-83-2-1425

ANALYTICAL CHEMISTRY LABORATORY
Test Results

Date May 11, 1983

For Jewell Finch
U.S.E.P.A.
MD-50
RTP, NC 27711

Analysis Organic Vapors

Sample Type Personal Monitors

Date Received 5/10/83

NSI #	070	071	072	073
MONITOR #	IW3401	IW3543	IW3433	IW3589
Nitrotoluene	*N.D.	N.D.	N.D.	N.D.
Isophorone	N.D.	N.D.	N.D.	N.D.
Tetrachlorotoluene	N.D.	N.D.	N.D.	N.D.
Triphenylmethane	N.D.	N.D.	N.D.	N.D.
2,4,6-triphenoxy-s-triazine	N.D.	N.D.	N.D.	N.D.

*N.D. = None Detected

Remarks Monitors were extracted with carbon disulfide. All four monitors had high background levels.

Analyst Zora E. Bunn Zora E. Bunn

AN EQUAL OPPORTUNITY EMPLOYER

NORTHROP SERVICES, INC. A Subsidiary of Northrop Corporation
ENVIRONMENTAL SCIENCES

P.O. Box 12313
Research Triangle Park, N. C. 27709
Telephone: (919) 549-0611

4240-83-2-1457

ANALYTICAL CHEMISTRY LABORATORY
Test Results

Date May 20, 1983

Analysis Organic Vapors

Sample Type Personal Monitors

Date Received 5/11/83

For Jewell Finch
U.S.E.P.A.
MD-50
RTP, NC 27711

NSI #	074	075	076	077
MONITOR #	IW3252	IW3234	IW3404	IX8581
Nitrotoluene	*N.D.	N.D.	N.D.	N.D.
Isophorone	N.D.	N.D.	N.D.	N.D.
Tetrachlorotoluene	N.D.	N.D.	N.D.	N.D.
Triphenylmethane	N.D.	N.D.	N.D.	N.D.
2,4,6-triphenoxy-s-triazine	N.D.	N.D.	N.D.	N.D.

*N.D. = None Detected

Remarks

Analyst Zora E. Bunn Zora E Bunn

AN EQUAL OPPORTUNITY EMPLOYER

NORTHROP SERVICES, INC. A Subsidiary of Northrop Corporation
ENVIRONMENTAL SCIENCES

P.O. Box 12313
Research Triangle Park, N. C. 27709
Telephone: (919) 549-0611

4240-83-2-1504

ANALYTICAL CHEMISTRY LABORATORY
Test Results

Date May 27, 1983

For Jewell Finch

U.S.E.P.A.

MD-50

Research Triangle Park, NC 27711

Analysis Organic Vapors

Sample Type Personal Monitors

Date Received 5/19/83

NSI #	082	083	084	085
Monitor #	IW3412	IX8649	IW3517	IX8530
nitrotoluene	*N.D.	*N.D.	*N.D.	*N.D.
isophorone	N.D.	N.D.	N.D.	N.D.
tetrachlorotoluene	N.D.	N.D.	N.D.	N.D.
triphenylmethane	N.D.	N.D.	N.D.	N.D.
2,4,6-triphenoxy~~-s~~-triazine	N.D.	N.D.	N.D.	N.D.

NSI #	086	087
Monitor #	IX8975	IX8552
tetrahydrofuran	N.D.	N.D.
methyl vinyl ketone	N.D.	N.D.

Remarks *N.D. = None Detected

Analyst

AN EQUAL OPPORTUNITY EMPLOYER

APPENDIX D

PROJECT CORRESPONDENCE

TRW ENVIRONMENTAL ENGINEERING DIVISION

T310.JFM.83.007
May 17, 1983

Mr. Dave Sanchez
U.S. EPA
M.D. 63
Research Triangle Park, NC 27711

Dear Mr. Sanchez:

At your request, TRW (Mr. Stackhouse and myself) collected wipe samples in specified areas around the incinerator in the wing G area of the National Environmental Research Center. The samples consisted of wipes of 1) tools immersed in #2 fuel oil, 2) two pieces of test apparatus, 3) a "decontaminated" wrench, 4) the top of a receptacle for contaminated materials, and 5) a sample of the #2 fuel oil used for tool cleaning. Because of analytical difficulties associated with high hydrocarbon background due to the presence of fuel oil, only the decontaminated wrench and receptacle wipes were analyzed.

The two wipe samples and a blank were immersed in 15 mL of methylene chloride and vigorously agitated for 5 minutes. The methylene chloride was transferred to graduated conical centrifuge tubes and reduced in volume to 0.5 mL. Two microliters of each sample were injected onto a fused silica SE-54 capillary column (25 meters x 0.25 mm) with detection by flame ionization. Pure standards of nitrotoluene, isophorone, chlorobenzotrichloride (tetrachlorotoluene), and triphenylmethane were obtained from Dr. Ray Merrill of EPA and prepared in methylene chloride. No peaks were observed at a detection limit of 15 ng for the retention times indicated by the standards.

If you have questions or need additional information, please do not hesitate to call. Thank you.

Sincerely,

J. F. McGaughey

J. F. McGaughey
Laboratory Services

JFM/dh

cc: J. Homolya
R. Jongleux
C. Stackhouse

Progress Center
3200 E. Chapel Hill Rd./Nelson Hwy.
P.O. Box 13000
Research Triangle Park, N.C. 27709 (919) 541-9100

TRW ENVIRONMENTAL ENGINEERING DIVISION

T310.JFM.83.009
June 22, 1983

Mr. Dave Sanchez
U.S. Environmental Protection Agency
Mail Drop 63
Research Triangle Park, North Carolina 27711

Dear Mr. Sanchez:

On May 26th of this year, I collected twelve (12) filter-wipe samples in the "Wing G" area of the National Environmental Research Center. They are as follows:

TRW Control #	Description
4772	Sampling Port on Stack
4773	Temperature probes
4774	Recycle Line
4775	Top of Drum, clean fuel
4776	Regulator on Vent
4777	Inside of Red flue gas line from cooler
4778	Probe Tip Nozzle
4779	Top of feed barrel
4780	Cooling water condensate
4781	Floor wipe at Bay Door to outside
4782	Area A
4783	Area C

The analytical procedure utilized was identical to that described in a previous letter-report sent to you and dated May 17, 1983.

Four of the samples contained detectable amounts of the marker compound, triphenylmethane. The surface area of the wiped areas were provided by Mr. Bob Jongleux. The results, which were previously conveyed to you, are given below:

TRW Control #	Total Micrograms	Surface Area, $ft.^2$	$Micrograms/ft.^2$
4774	179	1.05	170
4775	34.5	2.76	12.5
4779	910	2.76	330
4783	10.1	4.0	2.53

On June 16, Mr. C. Stackhouse of TRW, collected two (2) additional filter-wipe samples, one from the top of the "feed-barrel" (TRW #4792) and one from the walls of the "feed-shed" (TRW #4793).

Sample # 4793 (shed walls) showed no triphenylmethane at the level of 15 ng. However, sample #4792 (feed barrel) gave a positive response of 229 µg total or 83 µg/ft.^{2} In addition, this sample was analyzed by GC/MS and qualitatively identified as triphenylmethane.

If you have any questions, please do not hesitate to call me. Thanks.

Sincerely,

James F. McGaughey
Laboratory Services

JFM/mlc

cc: H. Dempsey - Accurex
R. Clayton - Accurex
J. Finch - EPA
R. Merrill - EPA
D. Wagoner - TRW
B. Stackhouse - TRW
R. Jongleux - TRW

APPENDIX E

FACILITY OPERATIONAL DATA - PHASE 1

TEST 1 - Run 1 Facility Operational Data

Time	T1-1	-2	-3	-4	-5	-6	-7	-12	T3-2	-3	-4	T3-5	T3-7	-9	T5-5	-6	Oil Flow gph	Dist. ΔP	F.B. Press	Bed Density ΔP	Bed Ht. ΔP	Bed ΔP	Bag House ΔP	Inlet Pres.	T3-1
1300	660	1793	1783	1787	1804	1808	1764	1715	1695	1678	1628	1352	1281	947	371	260	1.9	28	-2	37.5	42.5	40.0	2.2	76	410
1330	666	1792	1779	1786	1802	1810	1770	1720	1695	1678	1631	1359	1287	961	366	255	2.0	28	-2	37.5	42.5	40.0	2.2	76	410
1400	677	1809	1796	1803	1820	1826	1784	1732	1710	1693	1646	1367	1294	972	370	262	2.1	26	-2	37.5	42.5	40.0	2.1	76	408
1430	687	1809	1798	1805	1819	1824	1776	1727	1708	1692	1650	1375	1302	982	375	264	1.9	28	-2	37.0	42.0	40.0	2.2	76	410
Flow dropped to 1.6, Temp to 1782 @ 1450																									
1530	683	1792	1779	1787	1806	1812	1770	1731	1711	1694	1650	1376	1303	985	375	265	2.1	27.5	-1.9	37.0	42.0	39.5	2.2	76	413
1550	689	1798	1784	1793	1808	1817	1778	1741	1717	1700	1655	1382	1310	989	376	265	1.9	28	-2	37.0	42.0	39.5	2.2	76	412
1600	689	1798	1785	1792	1809	1814	1764	1722	1705	1692	1651	1388	1315	995	376	271	1.9	28	-1.8	37.0	42.0	39.5	2.2	76	412
1630	687	1795	1783	1793	1807	1816	1771	1730	1711	1690	1655	1391	1318	996	376	275	1.9	28	-1.8	37.0	41.5	39.0	2.2	76	411
1700	688	1791	1779	1788	1805	1813	1768	1729	1704	1692	1652	1394	1322	998	376	275	1.9	28.5	-1.8	36.5	41.5	39.0	2.2	76	412
Flame out 1725 Nozzle Plugged - Test Terminated																									
1730		1690	1672	1679	1681	1669																			
Avg.	684	1797	1785	1794	1810	1817	1773	1729	1707	1693	1648	1379	1307	985	373	267	1.95	27.8	-1.91	37.1	42.1	39.6	2.19	65	411

Inlet Orifice ΔP	O_2 %	THC %	CO	CO_2
2.2	12.5	0		
2.2	13.0	0	Test Start	
2.1	13.0	0		
2.2	13.6			
2.2	12.0			
2.2	13.2			
2.2	13.5			
2.2	-			
2.2	12.5			

All temperatures in °F. Pressures in inches of water unless noted.

TEST 1 - Run 2 Facility Operational Data

Time	T1-1	-2	-3	-4	-5	-6	-7	-12	T3-2	-3	-4	T2-7	-8	T3-5	-7	-9	T5-5	-6	Oil Flow gph	Dist. ΔP	F.B. Press	Bed Density ΔP	Bed ΔP	Bag House ΔP	Inlet ΔP	Inlet P	O_2 %	THC %	T
1035	536	1813	1806	1807	1815	1785	1690	1603	1587	1566	1512	1502	1439	1244	1179	850	336	241	2.2	28	2.2	34	36.5	1.5	-	72	12.5	<.1	326
1105	580	1816	1806	1809	1820	1808	1713	1620	1599	1578	1522	1513	1452	1254	1189	888	342	245	2.3	28.5	2.2	34	35.5	1.5	-	72	12.0	<.1	
1143	598	1807	1797	1797	1810	1786	1698	1608	1588	1579	1518	1508	1450	1256	1192	892	341	242	2.1	28.5	2.2	34	35.5	1.5	5	72	12.5	<.1	
1200	600	1806	1798	1798	1811	1791	1700	1616	1597	1758	1525	1516	1457	1260	1194	894	342	243	2.1	28	2.2	34	35.5	1.5	5	72	12.0	<.1	395
1230	611	1815	1808	1808	1821	1803	1723	1625	1605	1585	1532	1523	1466	1268	1201	899	341	244	2.1	27.5	2.2	34.5	36	1.5	5	72	11.9	<.1	395
1300	630	1813	1802	1805	1816	1799	1720	1626	1606	1588	1536	1527	1471	1273	1206	902	341	244	1.95	28	2.2	34.5	36	1.5	4.8	72	11.9	<.1	403
1330	641	1813	1804	1805	1818	1802	1722	1629	1611	1593	1543	1533	1478	1280	1212	906	345	245	1.95	28	2.2	35	36.5	1.5	4.8	72	12.0	<.1	400
1400	658	1813	1802	1804	1816	1802	1722	1634	1616	1597	1548	1539	1485	1287	1219	911	347	259	2.0	28	2.2	35	36.5	1.5	4.7	72	12.2	<.1	400
1430	664	1809	1796	1801	1812	1800	1729	1634	1617	1597	1550	1541	1489	1292	1224	916	350	251	2.0	27.5	2.2	35	36.5	1.5	4.5	72	12.3	<.1	405
1500	669	1809	1800	1803	1814	1803	1727	1636	1620	1604	1556	1546	1494	1298	1230	919	345	251	2.0	27.5	2.2	35	36.7	1.5	4.9	72	13.5	<.1	398
1530	675	1813	1805	1809	1821	1805	1727	1645	1626	1608	1563	1554	1502	1304	1236	924	346	252	1.9	27.5	2.2	35	37	1.5	4.8	72	13.5	<.1	
1600	671	1815	1805	1807	1821	1808	1833	1645	1631	1612	1565	1565	1503	1307	1239	927	354	255	1.85	26.5	2.2	35	37	1.5	4.5	72	14.6	<.1	393
											Stop Test - Clean Fuel 1602																		
Avg.	628	1812	1802	1804	1816	1799	1725	1627	1609	1590	1539	1531	1474	1277	1210	902	344	247	2.04	27.8		34.75	36.3	1.5	4.8				

All temperatures in $^{\circ}$F. Pressures in inches of water unless noted.

TEST 1 - Run 3 Facility Operational Data

Time	T1-1	-2	-3	-4	-5	-6	-7	-12	T3-2	-3	-4	T2-7	-8	T3-5	-7	-9	T5-5	-6	Oil Flow gph	Dist. ΔP	F.B. Press	Bed Density ΔP	Bed ΔP	Bag House ΔP	Inlet P	Inlet Orifice ΔP	O_2 %	THC %	CO ppm	CO_2 %	T3-1
945	637	1813	1802	1808	1827	1815	1750	1623	1608	1583	1526	1517	1456	1262	1203	926	342	247	2.3	29.5	<-2	33.5	37	1.5	72	5.2	11.1	<.1	25	6.3	414
1000	640	1816	1802	1812	1833	1821	1757	1624	1608	1585	1539	1521	1458	1258	1197	916	335	241	2.15	29.0	<-2	33.5	37	1.4	72	5.0	11.6	<.1	25	6.2	407
1030	642	1806	1798	1800	1818	1803	1724	1615	1596	1575	1522	1515	1456	1253	1190	904	330	237	2.0	29.0	<-2	33.5	37	1.4	70	4.8	12.5	<.1	25	5.5	407
1100	643	1810	1803	1805	1826	1817	1742	1630	1612	1593	1538	1528	1470	1259	1193	903	325	236	2.0	28.5	<-2	34	37	1.4	70	4.8	12.3	<.1	29	6.0	407
1130	648	1814	1805	1805	1828	1820	1741	1632	1614	1592	1549	1532	1475	1264	1198	904	330	235	1.9	27.5	<-2	34	37.5	1.4	70	4.5	12.7	<.1	29	5.4	400
1200	640	1810	1800	1801	1824	1813	1745	1636	1615	2597	1545	1538	1482	1269	1202	905	325	236	1.9	27.5	<-2	34.5	38	1.4	70	4.6	12.8	<.1	29	5.3	391
1230	646	1813	1806	1806	1824	1814	1749	1641	1622	1604	1553	1545	1490	1279	1209	911	335	235	1.8	27.5	<-2	34.5	38	1.4	70	5.0	12.9	<.1	17.5	5.8	402
1300	656	1816	1810	1809	1830	1818	1747	1646	1628	1610	1561	1552	1498	1286	1215	915	335	242	1.8	27.5	<-2	34.5	38	1.4	70	4.8	12.9	<.1	17.5	5.2	402
1330	654	1808	1803	1805	1822	1812	1745	1645	1625	1610	1561	1552	1500	1289	1220	917	334	240	1.8	27.5	<-2	34.5	38	1.4	70	4.8	13.2	<.1	17.5	5.2	404
1400	659	1816	1810	1810	1828	1815	1745	1649	1628	1613	1565	1556	1505	1293	1224	919	335	243	1.8	27.2	<-2	34.5	38	1.4	70	4.5	13.2	<.1	10	5.2	404
1430	661	1812	1804	1807	1822	1808	1745	1645	1627	1612	1565	1556	1506	1296	1223	921	336	243	1.75	27.5	<-2	34.5	38	1.4	70	4.5	13.5	<.1	15	4.9	406
1500	649	1807	1800	1802	1820	1803	1734	1646	1630	1615	1567	1558	1509	1298	1228	920	333	244	1.7	27.5	<-2	34.5	38	1.4	70	4.5	13.4	<.1	15	5.0	410
Avg.	648	1812	1804	1806	1825	1813	1744	1636	1618	1599	1548	1539	1484	1276	1209	913	333	240	1.903	28	<-2	34.2	37.6	1.4	70	4.75	12.68		19	5.5	405
1540	599	1970	1963	1962	1983	1996	1920	1788	1761	1729	1672	1655	1586	1317	1242																
1550		2000+	2000+	2000+	2000+	2000+																									

Pressure in fuel line was fairly high through run, 13-22 psi. After switching to clean fuel at end of run, value went to 22 psi with reduced flow rate

All temperatures in °F. Pressures in inches of water unless noted.

TEST 2 - Facility Operational Data

	Time	T1-1	-2	-3	-4	-5	-6	-7	-12	T3-2	-3	-4	T2-7	-8	T3-5	-7	-9	T5-5	-6	Oil Flow gph	Dist. ΔP	F.B. Press	Bed Density ΔP	Bed ΔP	Bag House ΔP	Inlet P	Inlet Orifice ΔP	O_2 %	THC %	CO ppm	CO_2 %	T3-1
	1015	621	1838	1820	1813	1818	1796	1716	1609		1565	1512	1501	1440	1229	1165	937	310	213	2.0	34.5	<-2	24	25	1.2	58	4.2	11.7	0	22	6.5	413
	1030	608	1830	1813	1817	1822	1796	1710	1615		1571	1518	1507	1445	1230	1163	850	326	231	1.9	34.5		23.5	25	1.4	58	4.2	11.8	0	22	6.3	412
Run 1	1100	606	1839	1824	1828	1827	1799	1717	1615	141	1574	1521	1511	1452	1236	1188	828	360	266	1.7	34		23.5	25	1.4	58	4.0	12.6	0	18	5.6	
	1130	597	1823	1810	1814	1816	1793	1720	1618	152	1578	1525	1515	1458	1240	1172	893	405	287	1.8	34		23.5	25	1.3	57	4.0	12.8	0	18	6.0	416
	1200	601	1833	1817	1823	1824	1798	1723	1625	169	1584	1535	1524	1466	1246	1172	908	455	317	1.75	34		24	25	1.3	57	4.0	12.7	0	20	6.0	414
	1230	606	1840	1823	1829	1829	1801	1725	1633	179	1592	1541	1533	1475	1253	1183	916	466	335	1.7	33.5		24	25	1.4	57	4.0	12.8	0	18	5.8	413
	1300	602	1834	1819	1825	1828	1806	1727	1636	182	1598	1546	1536	1481	1258	1187	913	475	340	1.65	33.5		24.5	25	1.4	58	3.8	12.7	0	15	6.1	406
Run [illegible]	1330																															
	1415	598	1822	1808	1814	1817	1793	1722	1638	188	1601	1552	1543	1490	1266	1195	924	492	350	1.65	33.5		24.5	25	1.4	58	4.8	12.9	0	15	5.7	406
	1445	802	1830	1815	1820	1821	1797	1721	1640	191	1603	1555	1547	1494	1272	1200	924	497	355	1.6	33.5		24.5	25	1.4	58	3.8	12.8	<.1	15	5.4	407
	1515	603	1836	1823	1826	1831	1805	1729	1649	193	1614	1566	1555	1504	1278	1206	934	495	355	1.6	33.5		24.5	25	1.5	58	3.8	13.2	<.1	15	5.7	404
	1545	603	1833	1818	1825	1824	1800	1722	1649	193	1614	1568	1558	1507	1282	1209	936	501	365	1.6	33.5		24.5	25	1.5	58	3.8	13.2	<.1	15	5.6	405
	1515	601	1833	1819	1825	1828	1803	1730	1653	195	1617	1572	1563	1511	1284	1211	935	503	365	1.55	33.5		24.5	25	1.5	58	3.8	13.4	<.1	15	5.7	406
Run [illegible]	1545	599	1834	1820	1826	1830	1806	1735	1658	198	1621	1576	1566	1516	1286	1213	934	505	366	1.5	33		24.5	25	1.5	57	3.8	13.2	<.1	15	5.6	406
	1715	596	1833	1817	1823	1826	1804	1736	1658	198	1622	1578	1566	1517	1288	1215	935	506	366	1.5	33		24.5	25	1.5	57	3.85	13.18	<.1	13	5.5	405
	Avg.	603	1832	1818	1822	1824	1800	1724	1653		1577	1548	1538	1483	1261	1190	912	450	322	1.68	33.6		24.1	25	1.4	57.	3.90	12.8	<.1	16.7	5.82	409

All temperatures in ^{o}F. Pressures in inches of water unless noted.

APPENDIX F

PROTOCOL FOR THE COLLECTION AND ANALYSIS OF VOLATILE POHCS USING VOST

United States
Environmental Protection
Agency

EPA-600/8-84-007
March 1984

EPA

Research and Development

PROTOCOL FOR THE COLLECTION AND ANALYSIS OF VOLATILE POHCs USING VOST

Prepared for

Office of Solid Waste and Emergency Response

Prepared by

Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

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EPA-600/8-84-007
March 1984

PROTOCOL
FOR THE
COLLECTION AND ANALYSIS OF
VOLATILE POHCs USING VOST

By

Earl M. Hansen

Envirodyne Engineers, Inc.
12161 Lackland Road
St. Louis, Missouri 63146

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EPA Project Officer: Robin M. Anderson
Technical Support Office
U. S. Environmental Protection Agency
Industrial Environmental Research Laboratory
Research Triangle Park, NC 27711

Prepared For

Office of Research and Development
U.S. Environmental Protection Agency
Washington, DC 20460

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FOREWORD

This report has been produced by EPA's Office of Research and Development as part of ongoing studies in support of engineering research and regulatory programs including EPA's Office of Solid Waste, EPA Regional Offices, and appropriate State Agencies. The document contains state-of-the-art operating protocols for sampling and analysis of volatile organic constituents of flue gases from hazardous waste incinerators or other similar combustor systems. It is intended as a reference to be used for guidance by personnel of the regulatory groups, personnel associated with engineering R&D, and the regulated community.

Publication of this report does not constitute official designation as an EPA method. Official test methods for hazardous waste related programs are published in SW-846 "Tests Methods for Evaluating Solid Waste," as well as in the Federal Register.

Frank T Princiotta

Frank T. Princiotta
Director
Industrial Environmental Research Laboratory

ABSTRACT

This document provides a state-of-the-art operating protocol for sampling and analysis of volatile organic constituents of flue gas from hazardous waste incinerators or other similar combustion systems using the Volatile Organic Sampling Train (VOST). It is intended to be used for guidance by personnel of the regulatory groups, personnel associated with engineering Research and Development, an dthe regulated community.

The VOST is designed to extract and concentrate volatile organic compounds (boiling ≤100°C) from stack gas effluents. The concentrated organics are analyzed by procedures chosen to be compatible with the VOST in order to obtain flue gas concentration levels. This information is necessary to perform destruction and removal efficiency (DRE) calculations for incinerator operations. The results of laboratory evaluation and field use of the VOST have shown that the VOST provides sufficient sensitivity to permit calculation of a DRE equal to or greater than 99.99 percent for volatile organics present in the waste feed at 100 µg/g.

The VOST is directly applicable to organic compounds with boiling points of 30° to 100°C. Many organic compounds with boiling points less than 30°C, or with boiling points in the 100° to 150°C range, may also be collected and analyzed by this method. Field application of the VOST for compounds with boiling points outside the 30° to 100°C range should be attempted only after laboratory evaluation of the collection and recovery efficiencies of the specific compounds.

The document is presented in two parts. Part A describes the key components of the train, the procedures for preparation of the sorbent materials, and procedures for sample collection using the VOST. Part B describes the procedures for analysis of VOST sorbent cartridges for volatile principal organic hazardous constituents (POHCs) using purge-trap-desorb gas chromatography/mass spectrometry (P-T-D GC/MS). Quality control procedures are presented in both Sections A and B.

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INTRODUCTION

The Resource Conservation and Recovery Act (RCRA) requires that owners/operators of facilities which treat hazardous waste by incineration ensure that the incinerators are operated in a manner which does not endanger human health or the environment (1,2). The Code of Federal Regulation, Title 40, Part 264, requires that a destruction and removal efficiency (DRE) of 99.99 percent be achieved for each principal organic hazardous constituent (POHC) designated in the Trial Burn Permit (3). The DRE standard implicitly requires sampling and analysis to quantify POHCs in the waste feed material and stack gas effluent. The "Sampling and Analysis Methods for Hazardous Waste Combustion" manual (method manual) provides information on methods that are applicable for collection and analyses of POHCs in process streams of hazardous waste incinerator units (4).

The methods manual identifies three possible methods for the collection of volatile organics (those with boiling points <100°C). The methods include bag, glass bulb, and the Volatile Organic Sampling Train (VOST). Evaluation of the bags and bulbs indicates that these sampling systems are subject to a number of technical problems. The most important of these problems is the inadequate sensitivity for detection of POHCs present in low concentrations.

The VOST provides increased sensitivity to low level concentration of volatile POHCs due to the ability to concentrate the gaseous effluent. The results of laboratory evaluation and field application of the VOST have shown that it provides sufficient sensitivity to permit calculation of a DRE equal to or greater than 99.99 percent for volatile POHCs which are present in the waste feed at 100 µg/g (5).

The methods manual identifies the VOST as a suitable sampling system for volatile organics and includes a paper describing the VOST (5). A detailed protocol was not included in the methods manual due to the fact that this is outside the scope of the document. Since the VOST is new technology, it is felt a protocol should be made available.

The purpose of this protocol is to provide a standard operating procedure to users of the VOST in the collection and analysis of samples for volatile POHCs in the gaseous effluents of hazardous waste incinerators or gaseous effluents of hazardous waste co-fired combustion processes. The protocol is presented in two parts. Part A describes the key components of the VOST train, and the procedure for sample collection using VOST. Part B describes the procedures for analysis of VOST sorbent cartridges for volatile POHCs using purge-trap-desorb gas chromatography/mass spectrometry (P-T-D GC/MS).

PART A

SAMPLE COLLECTION FOR THE DETERMINATION OF THE EMISSIONS OF VOLATILE ORGANIC COMPOUNDS FROM HAZARDOUS WASTE INCINERATORS

1. SCOPE AND APPLICABILITY

1.1 This protocol describes the method of collection of volatile principal organic hazardous constituents (POHCs) from the stack gas effluents of hazardous waste incinerators. This method is applicable to compounds with boiling points in the range of 30° to 100°C. If the boiling point of a POHC of interest is less than 30°C, the POHC may breakthrough the sorbent under the conditions of the sample collection procedure. Field application for POHCs of this type should be supported by laboratory data which demonstrates the efficiency of a volatile organic sampling train (VOST) to collect POHCs with boiling points less than 30°C (Part B, Section 7). The collection of organic compounds with low boiling points may require using reduced sample volumes collected at flow rates between 250 and 500 milliliters per minute (ml/min). Many compounds which boil in the range of 100° to 150°C (e.g., chlorobenzene, ethylbenzene, tetrachloroethane, bromoform) may be efficiently collected and analyzed using this method. VOST recovery efficiencies for these compounds should also be demonstrated, where necessary, by laboratory data of the type described above (Part B, Section 7).

1.2 This method is applicable to the determination of volatile POHCs in the stack gas effluent of hazardous waste incinerators and other similar combustion systems. This method is designed for use in calculating the destruction and removal efficiency (DRE) for the volatile POHCs. The method provides sufficient precision and accuracy to enable the calculation of DRE values equal to or greater than 99.99 percent (5).

1.3 The sensitivity of this method is dependent upon the level of interferences in the sample and the presence of detectable levels of volatile POHCs in blanks. The target detection limit of this method is 0.1 micrograms per cubic meter ($\mu g/m^3$) [0.1 nanograms per liter (ng/l)] of flue gas which permit calculation of a DRE equal to or greater than 99.99 percent for those volatile POHCs which may be present in the waste feed stream at 100 parts per million (ppm). Laboratory development data (5) have demonstrated a range of 0.1 to 100 $\mu g/m^3$ (0.1 to 100 ng/l) for selected volatile POHCs.

1.4 The range of applicability for this method is limited by breakthrough of volatile POHCs on the sorbent cartridges used to collect the sample, caution should be exercised in using the VOST to collect samples from a stack gas stream in which one or more of the volatile POHCs are present in concentrations greater than 500 $\mu g/m^3$ (500 ng/l). If, for example, an incinerator uses a waste fuel containing volatile POHCs at a concentration of 50,000 to 100,000 µg/g, and the incinerator achieves a DRE of 99.99%, then the concentration in the effluent would be 500 to 1000 µg/m^3 (500 to 1,000 ng/l), respectively. Analysis using the procedures described in Part B of this method would probably result in overloading the analytical instrumentation and therefore would not provide valid data for that particular POHC. Where the waste contains volatile POHCs at high concentrations, consideration should be given to collecting gaseous effluent samples using SLOW-VOST techniques, gas bags or evacuated glass bulbs. Further, if the waste contains volatile POHCs at high (50,000 µg/g) and low (100 µg/g) concentrations, these POHCs might not be quantifiable using only VOST. In this case, consideration should be given to either collecting gaseous effluent samples using two VOST trains (one of these trains should be operated under conditions of SLOW-VOST), or using one VOST train (SLOW-VOST) and gas bags or glass bulbs. The examples cited above may result in chromatographic interferences or instrument overloading which will invalidate the results. If this situation is anticipated, laboratory evaluation should be conducted prior to field use of the VOST (Part B, Section 7).

1.5 This method is recommended for use only by experienced sampling personnel and analytical chemists, or under close supervision by such qualified persons.

2. SUMMARY OF METHOD

2.1 A 2-hour sample of gaseous effluent is collected on cartridges using six pairs of cartridges, each pair sampling a maximum of 20 liters of gaseous effluent. The samples are collected at a sampling flow rate of 1 liter per minute, using temperature controlled quartz or glass lined probe and a VOST. The gas stream is cooled to 20°C by passage through a water-cooled condenser and volatile POHCs are collected on a pair of sorbent cartridges. Liquid condensate is collected in an impinger placed between the two sorbent cartridges. The first sorbent cartridge (front cartridge) contains approximately 1.6 grams Tenax, and the second cartridge (back cartridge) contains approximately 1 gram each of Tenax and petroleum based charcoal (SKC Lot 104 petroleum base or equivalent, see Section 6.2.1), 3:1 by volume.

2.2 An alternative set of conditions for sample collection has been used. This method involves collecting a sample volume of 20 liters or less at a reduced flow rate. Operation of the VOST under these conditions has been referred to as SLOW-VOST. This method has been used to collect 5 liters of sample (0.25 liters/min for 20 minutes) or 20 liters of sample (0.5 liters/min for 40 minutes) on each pair of sorbent cartridges. Smaller sample volumes collected at lower flow rates should be considered when the boiling points of the POHCs of interest are below 30°C or for volatile POHCs present in high concentrations in the stack gas.

2.3 The gaseous effluent shall be sampled over a 2-hour period. This is accomplished using six pairs of Tenax and Tenax/charcoal cartridges sampling a maximum of 20 liters of gaseous effluent on each pair of cartridges. Fewer pairs of cartridges may be required using SLOW-VOST (Section 2.2). A minimum of three pairs of Tenax and Tenax/charcoal cartridges shall be collected for SLOW-VOST sampling.

2.4 Analysis of the cartridges (described in Part B) for volatile POHCs using purge-trap-desorb gas chromatograph/mass spectrometer (P-T-D GC/MS) which is carried out by thermally desorbing each cartridge with the gas passing through a water filled purge column onto an analytical trap. The analytical trap is subsequently heated and the effluent gas passes into the GC/MS.

3. PRECISION AND ACCURACY

3.1 The results of laboratory evaluations of the VOST for selected volatile POHCs showed that the recovery of the analytes from three pairs of replicate cartridges ranged from ±50 percent of the expected value (5).

3.2 Prior to field operation of the VOST at a hazardous waste incinerator, a laboratory trial should be conducted using either selected volatile POHCs of interest or two or more of the volatile POHCs for which data are available (5). The user shall demonstrate proficiency which is within the precision and accuracy of the method (Part B, Section 7). Experienced users of VOST who have demonstrated proficiency with the VOST and can provide data which supports the applicability of the VOST for the POHCs may be exempt from this requirement. Before the VOST shall be used to sample compounds below 30°C, the user must demonstrate through laboratory evaluations of the collection and recovery efficiencies that the accuracy and precision requirements are met (Part B, Section 7).

4. INTERFERENCES

4.1 Interferences arise primarily from background contamination of sorbent cartridges prior to or after use in sample collection. Many potential interferences can be due to exposure of the sorbent materials to solvent vapors prior to assembly and exposure to significant concentrations of volatile POHCs in the ambient air at hazardous waste incinerator sites. Benzene and toluene appear to be inherent contaminants on Tenax. This may present problems in the analysis of these compounds using VOST due to high background concentrations. To minimize this problem, the use of additional field blanks is recommended (see Section 8.5).

4.2 To avoid or minimize the low level contamination of train components with volatile POHCs, care should be taken to avoid contact of all interior surfaces or train components with synthetic organic materials (e.g. organic solvents, lubricating and sealing greases), and train components should be carefully cleaned and conditioned according to the procedures described in Sections 5, 6 and 7 of this protocol. The sorbent cartridges should be conditioned and stored in an environment free of volatile organic compounds.

5. APPARATUS

5.1 Volatile Organic Sampling Train

5.1.1 A schematic diagram of the principal components of the VOST is shown in Figure 1. A diagram of the assembled components of one version of the VOST is shown in Figure 2. The VOST consists of: a quartz or glass lined probe with a glass wool particulate plug, an isolation valve, a water cooled glass condenser with thermocouple placed at the outlet to monitor gas stream temperature (see Section 5.6.2), a sorbent cartridge containing Tenax (1.6 grams), an empty impinger for condensate removal, a second water cooled glass condenser, a second sorbent cartridge containing Tenax and petroleum based charcoal (3:1 by volume; approximately 1 gram of each), a silica gel drying tube, a calibrated rotameter, a sampling pump, and a dry gas meter.

5.1.2 The gas pressure during sampling and for leak checking is monitored by vacuum gauges which are in line with and downstream of the silica gel drying tube.

5.2 Probe

5.2.1 The probe shall be maintained at a temperature of at least 130°C in the gas stream prior to the first condenser. If one or more of the volatile POHCs boil between 130° to 150°C, the probe shall be maintained at a temperature equivalent to the boiling point of the least volatile POHC. The probe may require heating to achieve this temperature. If stack temperatures are excessively high, an air or water cooled probe may be required to

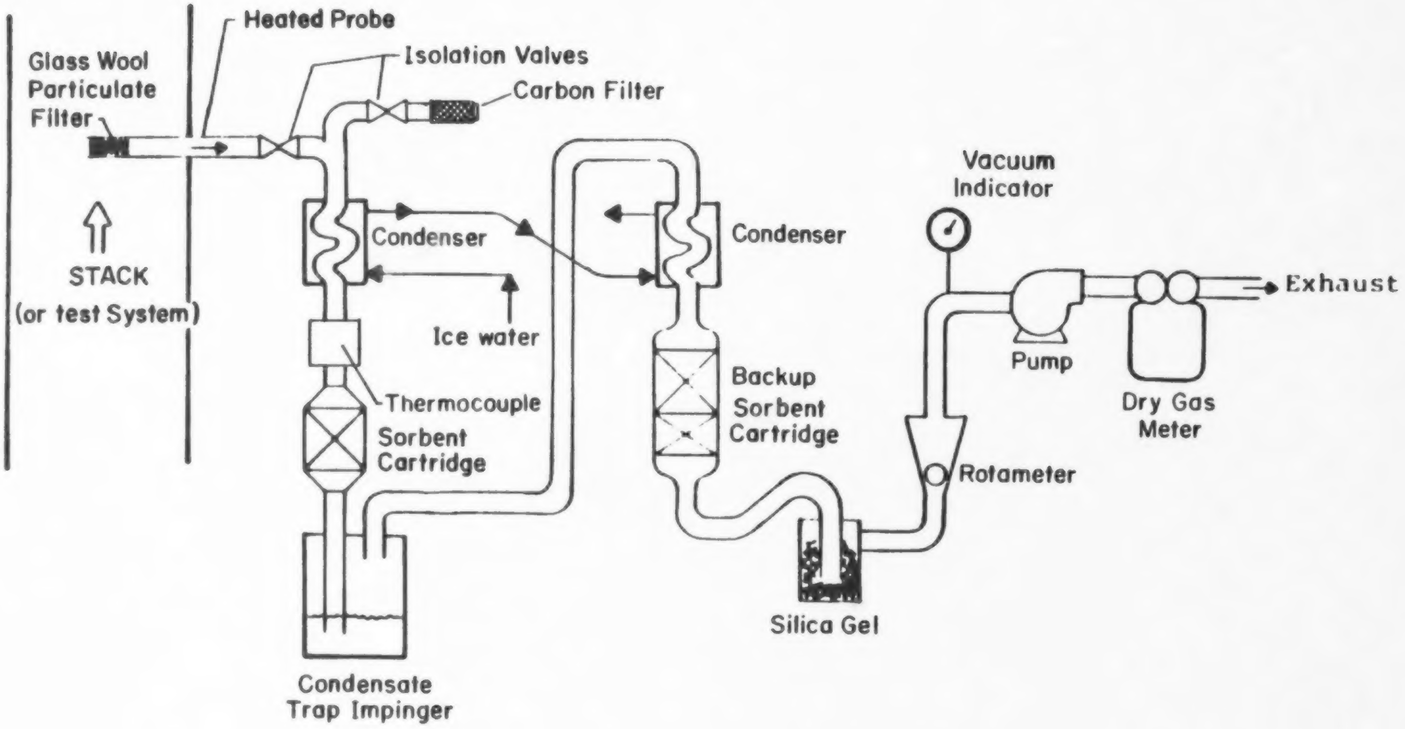

Heated Probe
Glass Wool Particulate Filter
Isolation Valves
Carbon Filter
STACK (or test System)
Condenser
Ice water
Thermocouple
Sorbent Cartridge
Condensate Trap Impinger
Condenser
Backup Sorbent Cartridge
Silica Gel
Vacuum Indicator
Rotameter
Pump
Dry Gas Meter
Exhaust

FIGURE I
SCHEMATIC OF
VOLATILE ORGANIC SAMPLING TRAIN
(VOST)

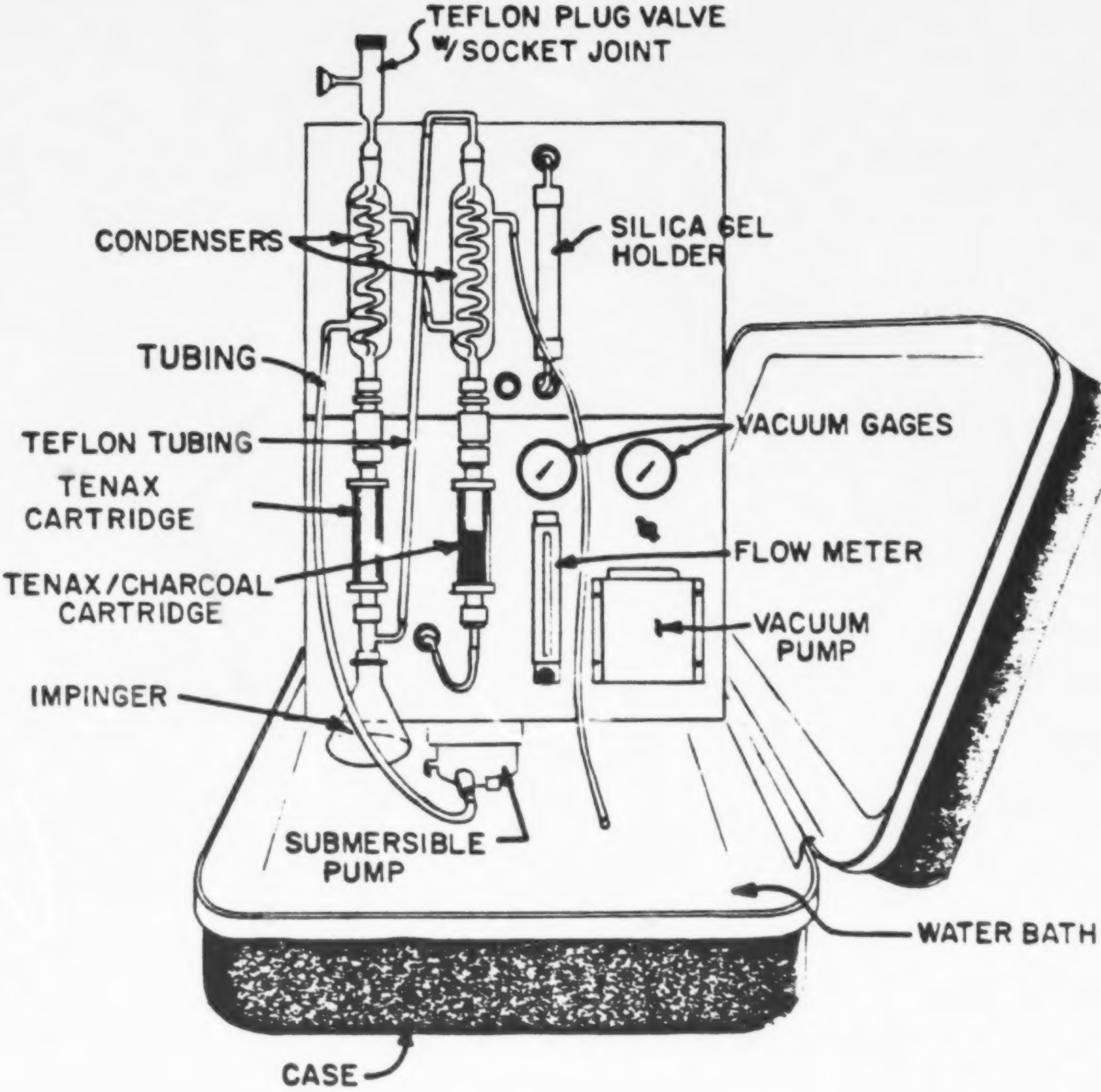

TEFLON PLUG VALVE
W/SOCKET JOINT
CONDENSERS
SILICA GEL
HOLDER
TUBING
VACUUM GAGES
TEFLON TUBING
TENAX
CARTRIDGE
FLOW METER
TENAX/CHARCOAL
CARTRIDGE
VACUUM
PUMP
IMPINGER
SUBMERSIBLE
PUMP
WATER BATH
CASE

FIGURE 2

VOLATILE ORGANIC SAMPLING TRAIN
(I/O CARTRIDGE DESIGN)

avoid damage to the probe and to achieve a temperature of 20°C at inlet to first sorbent cartridge (see Section 5.4). Isokinetic sample collection is not a requirement for the use of VOST since the compounds of interest are in the vapor phase at the point of sample collection.

5.3 Isolation Valve

5.3.1 The isolation valves must be a greaseless stopcock with a glass bore and sliding Teflon plug with Teflon wipers (Ace 8193 or equivalent). These valves are used to permit ambient air to be admitted after leak checking (Section 8.3) and for purging the probe prior to sample collection (Section 8.4).

5.4 Condensers

5.4.1 The condensers (Ace 5979-14 or equivalent) shall be of sufficient capacity to cool the gas stream to 20°C or less prior to passage through the first sorbent cartridge. The top connection of the condenser shall be able to form a leak-free, vacuum-tight seal without using vacuum sealing greases.

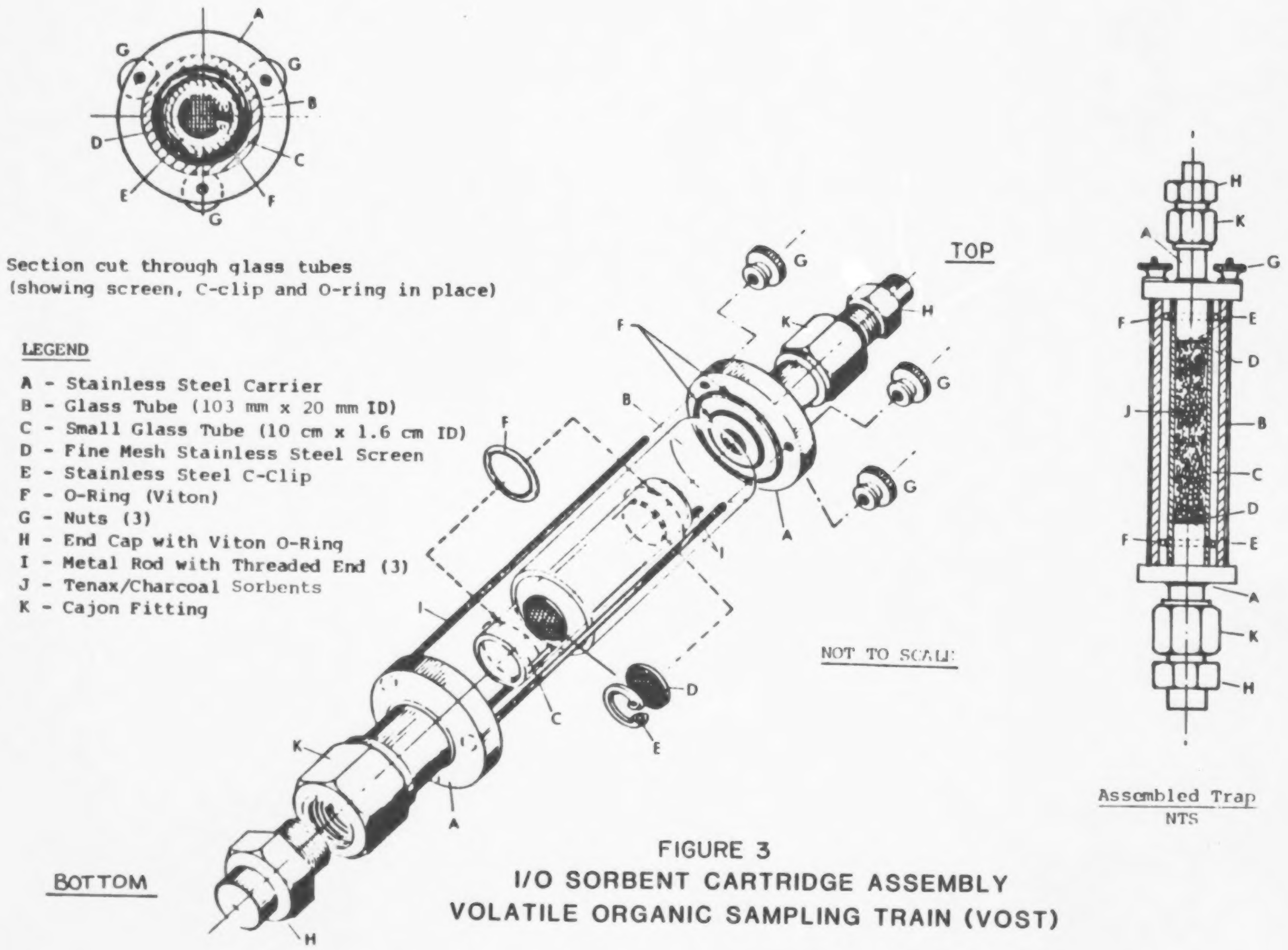
5.5 Sorbent Cartridges

5.5.1 The sorbent cartridges for the VOST are available and acceptable in either of two configurations: the inside/outside (I/O) or inside/inside (I/I) configuration. The terms I/O or I/I pertain to the instrument configuration for thermal desorption of the sorbent tubes. For the I/O design, the carrier gas contacts the inside and outside of the sorbent tubes during the desorption.

while for the I/I design, the carrier gas is directed only through the inside of the cartridge. For the I/O configuration, the cartridge is held in the VOST within an outer glass tube and in a metal carrier (Figure 3). For the I/I configuration, a single glass tube is used, with or without a metal carrier (Figure 4). In either case, the sorbent packing will be the same. The first pair of sorbent cartridges shall be packed with approximately 1.6 grams Tenax GC resin, and the second cartridge of a pair shall be packed with Tenax GC and petroleum based charcoal (3:1 by volume; approximately 1 gram of each).

5.5.2 The second sorbent cartridge shall be packed so that the sample gas stream passes through the Tenax layer first and then through the charcoal layer. Note that when sorbent cartridges are analyzed using the P-T-D GC/MS procedure described in Part B, Section 10.2, the gas flow through the sorbent cartridges during desorption is in the opposite direction from the gas flow through the sorbent cartridges during sample collection. The inlet side of each cartridge during sample collection shall be clearly identified so that it becomes the outlet side during the desorption step of the analytical procedure.

5.5.3 The sorbent cartridges shall be glass tubes with approximate dimensions of 10 centimeters by 1.6 centimeters (cm) I.D. The two acceptable designs (I/O, I/I) for the sorbent cartridge are described in further detail below.


A
G
G
B
D
C
E
F
G
TOP
G
G
G
H
K
F
B
F
A
I
I
NOT TO SCALE
D
C
E
K
A
BOTTOM
H
H
K
A
G
F
E
D
J
B
C
D
F
E
A
K
H

Section cut through glass tubes
(showing screen, C-clip and O-ring in place)

LEGEND

A - Stainless Steel Carrier
B - Glass Tube (103 mm x 20 mm ID)
C - Small Glass Tube (10 cm x 1.6 cm ID)
D - Fine Mesh Stainless Steel Screen
E - Stainless Steel C-Clip
F - O-Ring (Viton)
G - Nuts (3)
H - End Cap with Viton O-Ring
I - Metal Rod with Threaded End (3)
J - Tenax/Charcoal Sorbents
K - Cajon Fitting

Assembled Trap
NTS

FIGURE 3
I/O SORBENT CARTRIDGE ASSEMBLY
VOLATILE ORGANIC SAMPLING TRAIN (VOST)

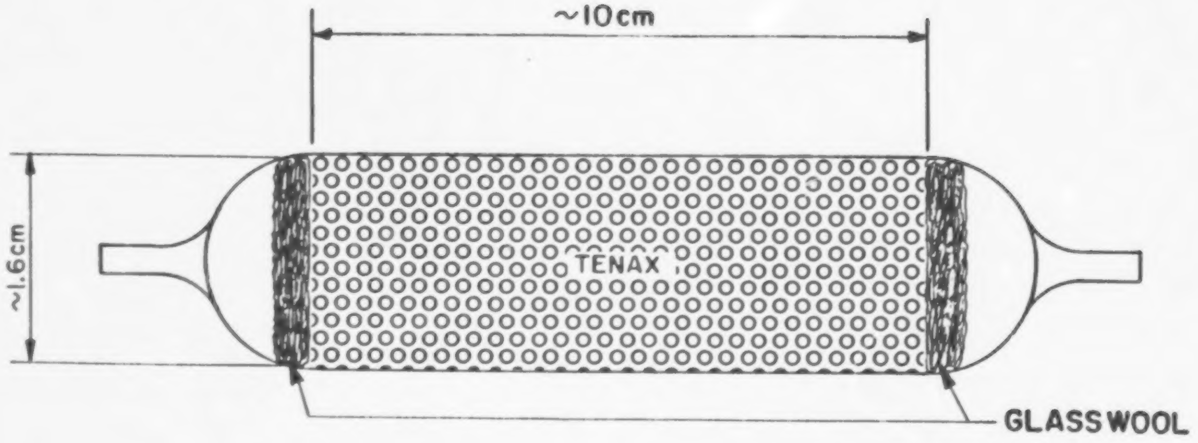

~10cm
~1.6cm
TENAX
GLASSWOOL

FIGURE 4

INSIDE - INSIDE VOST CARTRIDGE

5.5.3.1 Inside/Outside Type Sorbent Cartridge - A diagram of an I/O type sorbent cartridge is shown in Figure 3. In this design, the sorbent materials are held in the glass tube with a fine mesh stainless steel screen and a C-clip. The glass tube is then placed within a larger diameter glass tube, and held in place using Viton O-rings. The purpose of the outer glass tube is to protect the exterior of the resin-containing tube from contamination. The two glass tubes are held in a stainless steel cartridge holder. The ends of the glass tubes are held in place by Viton O-rings placed in machine grooves in each metal end piece. The three cylindrical rods are secured in one of the metal end pieces and fastened to the other end piece by appropriately sized nuts, thus sealing the glass tubes into the cartridge holder. The end pieces are fitted with a threaded nut onto which a threaded end cap, fitted with a Viton O-ring seal, is placed to protect the resin from contamination during transport and storage.

5.5.3.2 Inside/Inside Type Sorbent Cartridge - A diagram of an I/I type sorbent cartridge is shown in Figure 4. This cartridge is a single glass tube (10 cm by 1.6 cm I.D.) which has the ends reduced in size to accommodate a 1/4 or 3/8-inch Swagelok or Cajon gas fitting. The resin is held in place by glass wool at each end of the resin layer. The amounts of each type of sorbent material used in the I/I design are the same as for the I/O design. Threaded end caps are placed on the sorbent cartridge after packing with sorbent to protect the sorbent from contamination during storage and transport. Care should be exercised in the placement of the threaded end cap; the male threaded fitting should be backed about 1 mm from the end of the tube prior to tightening to prevent crushing or chipping of the end of the tube.

5.6 Impinger

5.6.1 The impinger which is used to collect aqueous condensate shall have a liquid volume of 125 ml and shall be capable of providing a leak-free seal to the outlet of the Tenax sorbent cartridge. The silica gel drying tube should have sufficient capacity for at least 20 to 30 grams of silica gel.

5.7 Metering System

5.7.1 The metering system for VOST shall consist of: vacuum gauges, a leak-free pump (Thomas Model 107 or equivalent), a calibrated rotameter (Linde Model 150 or equivalent) for monitoring the gas flow rate, a dry gas meter with 2 percent accuracy (7) at the required sampling rate, and related valves and equipment. The dry gas meter shall indicate 1 liter of gas volume for each revolution of the dial. Users of VOST have suggested that the rotameter does not need to be calibrated, but a calibrated rotameter serves as a useful verification of the volume measurement from the dry gas meter.

5.7.2 Provisions will be made for monitoring the temperature of the sample gas stream between the first condenser and first sorbent cartridge. This can be done by placing a thermocouple on the exterior glass surface of the outlet from the first condenser. The temperature at that point shall be less than 20°C. If it is not, an alternative system, providing increased cooling capacity, must be used.

5.8 Sample Transfer Lines

5.8.1 All sample transfer lines connecting the probe to VOST shall be less than 5 feet in length, if possible, and shall be heat-traced Teflon (to maintain a temperature of at least 130°C in the gas stream) with connecting fittings which are capable of forming leak-free, vacuum-tight connections without the use of sealing grease. All other sample transfer lines used with the VOST (e.g., to connect impinger with second condenser) will be Teflon with connecting fittings that are capable of forming leak-free, vacuum-tight connections without the use of sealing grease.

6. REAGENTS AND MATERIALS

6.1 Tenax, 35/60 Mesh (2,6-Diphenylene Oxide Polymer)

6.1.1 The new Tenax is Soxhlet extracted for 24 hours with methanol (Burdick & Jackson, pesticide grade or equivalent). The Tenax is dried for 6 hours in a vacuum oven at 50°C before use. Users of I/O and I/I sorbent cartridges have used slightly different thermal conditioning procedures. Although strict equivalence of the procedure has not been demonstrated, these methods have produced results that meet method quality assurance requirements (Part B, Section 7.4). I/O sorbent cartridges packed with Tenax are thermally conditioned by flowing organic-free nitrogen

30 ml/min through the resin while heating to 250°C for 8 hours. Sorbent cartridges of the I/I design packed with Tenax are thermally conditioned for 12 to 16 hours by flowing organic-free nitrogen (30 ml/min) through the resin while heating to 190°C. After thermal conditioning, the sorbent cartridges shall be stored for 24 hours at room temperature prior to monitoring the cartridges for residual organic contamination (see Section 7.4). The use of pentane to extract new Tenax and charcoal to remove nonpolar impurities is not recommended. (Users have experienced problems with residual pentane in the sorbents during analysis, and therefore, this procedure is not recommended.)

6.1.2 If very high concentrations of organic compounds have been collected on the resin (e.g., micrograms of analytes), the sorbent may require Soxhlet extraction as described above. Previously used Tenax cartridges are thermally reconditioned by the method described above.

6.2 Charcoal (SKC Lot 104 Petroleum Base or Equivalent)

6.2.1 New charcoal is prepared as described in 6.1.1 above. Due to possible problems with charcoal contamination, new charcoal should be used. Users of VOST have restricted the types of charcoal used in sorbent cartridges to only petroleum-based types. Criteria for other types of charcoal are currently under development. Other types of charcoal are acceptable if recovery of POHC in laboratory evaluations meet the criteria (±50 percent) of the method (Part B, Section 7).

6.3 Viton O-Ring

6.3.1 All O-rings used in VOST shall be Viton. Prior to use, these O-rings shall be thermally conditioned in a vacuum oven at 200°C for 48 hours. O-rings will be stored in clean, screw capped glass containers prior to use.

6.4 Glass Tubes/Condensers

6.4.1 The glass resin tubes and condensers shall be cleaned with a non-ionic detergent in an ultrasonic bath, rinsed well with organic-free water and dried at 110°C. Resin tubes of the I/O design should be assembled as described in Section 7 prior to storage. Resin tubes of the I/I design shall be stored in glass containers with Teflon lined screw caps. Condensers shall be capped with appropriate end caps prior to use.

6.5 Metal Parts

6.5.1 The metal parts (e.g., stainless steel carriers, C-clips, end plugs), used in either the I/O or I/I cartridge design, are cleaned by ultrasonication in a warm non-ionic detergent solution, rinsed with reagent water, air dried and heated in a muffle furnace for 2 hours at 400°C.

6.6 Silica Gel - - Indicating Type, 6-16 Mesh

6.6.1 New silica gel may be used as received. Silica gel which has been previously used shall be dried for 2 hours at 175°C.

6.7 Crushed Ice

6.7.1 Crushed ice from any source may be added to the coolant in the reservoir.

6.8 Water

6.8.1 Water used for leak checking resin cartridges and rinsing glassware shall be reagent (see Part B, Section 5.1); water used for cooling train components in the field may be tap water.

6.9 Glass Wool

6.9.1 Glass wool shall be Soxhlet extracted for 8 to 16 hours using pentane, and oven dried at 110°C before use. The glass wool shall be checked prior to use to ensure that it does not contain residual pentane. If all pentane cannot be removed, an alternate solvent such as methanol may be used.

6.10 Nitrogen

6.10.1 Nitrogen gas will be organic-free (Linde-Union Carbide, 99.999% pure, hydrocarbon-free, or equivalent). In-line moisture traps, 5A° molecular sieve adsorbent tubes, or cryogenic traps may be used to ensure purity.

7. ASSEMBLY AND CONDITIONING OF VOST SORBENT CARTRIDGES

7.1 Introduction

7.1.1 This section describes assembly of the sorbent cartridges and procedures for storage and transport of assembled cartridges.

The assembly and packing of the sorbent cartridges should be carried out in an area free of volatile organic material. This is preferably done in a laboratory in which no organic solvents are handled or stored, and in which the laboratory air is charcoal filtered. Alternatively, the assembly procedure can be conducted in a glove box or glove bag which can be purged with organic-free nitrogen.

7.2 Assembly of Tenax Cartridges

7.2.1 The Tenax, glass tubes and metal cartridge parts are cleaned and stored according to the procedures described in Section 6. Approximately 1.6 grams of Tenax is weighed and packed into the sorbent tube which has a stainless steel screen and C-clips (I/O design) or by glass wool (I/I design) in the downstream end. The Tenax is held in place by inserting a stainless steel screen and C-clips in the upstream end (I/O design) or glass wool (I/I design). The cartridges are assembled in the metal holders (Section 5.5) and leak checked by putting on one of the end caps and pressurizing the cartridge to 30 psi with organic-free nitrogen and immersing the cartridge in distilled water to check for the appearance of bubbles. Cartridges which are determined to be leak-free are then conditioned as described in Section 7.4.

7.2.2 Assembled and conditioned resin tubes of the I/O design are placed in ice water for storage and transport. Conditioned resin tubes of the I/I design are capped and placed in ice water for storage and transport. Limited experience by VOST users for storing and transporting resin tubes of the I/I design indicates that these tubes may be stored and transported in screw capped culture tubes placed in a clean glass or metal container which contains clean charcoal. The storage container is kept cold using ice or cold pack storage. Storage containers shall be kept in an area free from sources of organic contamination. The need for these precautions is dependent upon the user's ability to maintain blank cartridges free from contamination during storage and transport.

7.3 Assembly of Tenax/Charcoal Cartridges

7.3.1 The Tenax, charcoal and metal cartridge parts are cleaned and stored according to the procedures described in Section 6. The tubes are packed with approximately a 3:1 volume ratio of Tenax and charcoal (approximately 1 gram of each). The cartridge is assembled such that the inlet side during sampling collection is Tenax, followed by a layer of charcoal at the outlet side of the sorbent cartridge. The Tenax and charcoal are held in place by the stainless steel screens and C-clips (I/O design) or by glass wool (I/I design). The glass tubes of the I/O design containing the Tenax and charcoal are then leak checked as described in Section 7.2, checked for quality assurance as described in Section 7.4, and placed in the metal carriers according to the procedures outlined in Section 5.5. The end caps are placed on the assembled cartridges, and the assembled cartridges are placed in ice water for storage and transport as described in Section 7.2. Glass tubes of the I/I design are leak checked as described in Section 7.2, and conditioned as described in Section 7.4. The end caps are placed on the assembled cartridges, and they are placed in a suitable container for storage and transport as described in Section 7.2.

7.4 Sorbent Cartridge Quality Assurance

7.4.1 This section describes two methods which can be used to verify that the sorbent cartridges (Tenax and Tenax/charcoal) are free from background contamination prior to sample collection.

7.4.2 The gas chromatograph with flame ionization detector is calibrated using direct injection of propane standards at the appropriate concentrations. The chromatographic column shall be 1.8 m x 0.25 cm I.D., stainless steel or glass packed with 1% SP-1000 on Carbopack (60/80 mesh) or equivalent. Following assembly and leak checking of the sorbent cartridges, they are connected to a source of organic-free nitrogen. Nitrogen is passed through each trap at a flow rate of at least 30 ml/min, while the traps are heated to 200°C. The effluent from the trap is monitored with a flame ionization detector to check for emission of volatile organic compounds from the cartridge. The conditioning is continued until the amount of total chromatographable organics is below 0.2 ng as propane.

7.4.3 The gas chromatograph with flame ionization detector is calibrated by analyzing a Tenax cartridge which has been spiked with 175 ng benzene, 175 ng toluene and 150 ng bromofluorobenzene (BFB). The cartridge is spiked using the solvent-flush technique (see Part B, Section 6.2.3). The chromatographic conditions shall be the same as those described in Part B, or shall demonstrate the capability of identifying background levels of volatile POHCs with the required sensitivity. Following assembly and leak checking, the sorbent cartridge(s) is placed in the desorption heater for 10 minutes with a helium flow of 50 ml/min. The effluent from the cartridge is passed through a trap filled with glass beads which is cooled in liquid nitrogen. (If a pair of Tenax and Tenax/charcoal cartridges are monitored in a single desorption run, the Tenax/charcoal cartridge should be upstream ofthe Tenax cartridge.) At the end of the 10-minute desorption period, the glass bead filled trap is heated, the carrier gas flow directed to a gas chromatograph equipped with flame ionization detector and chromatographic column suitable for volatile organic analysis (see Part B, Section 4.3.2). Blank sorbent cartridges which are monitored using this technique shall contain less than 2 ng total chromatographable organics (as benzene or toluene). If in using this procedure the background exceeds 10 ng (as benzene or toluene), the cartridges must be recleaned and reanalyzed.

8. SAMPLE COLLECTION PROCEDURE

8.1 Pretest Preparation

8.1.1 All train components shall be cleaned and assembled as described in Sections 5, 6, and 7. The dry gas meter shall have been calibrated within 30 days prior to use. All field data shall be recorded on a Field Data Sheet (or field notebook). An example of a Field Data Sheet for VOST is shown in Figure 5.

8.2 VOST Assembly

8.2.1 The VOST is assembled according to the schematic diagram in Figure 2. Cooling water is circulated to the condensers and the temperature of the cooling water shall be maintained near 0°C. The end caps of the sorbent cartridges will be placed in a clean screw capped glass container during sample collection. The condensate pot shall not be immersed in ice water.

8.3 Leak Checking

8.3.1 The train is leak checked by closing the valve at the inlet to the first condenser and pulling a vacuum equivalent to 10 inches Hg (250 mm Hg) above the system's operating pressure. The traps and condensers are isolated from the pump and the leak rate noted. The system shall hold at 10 inches Hg above the system operating pressure with no discernible leakage (pressure increase of less than 0.1 inches Hg/min). If the leak check does not meet specifications, the cause of the problem must be identified and corrected and leak check repeated. After leak checking, the train should be returned to atmospheric pressure by attaching a charcoal filled tube to the isolation valves above the first condenser and opening the valve to admit air to the train through the charcoal filter. These procedures will minimize contamination of VOST train components by excessive exposure to the fugitive emissions at hazardous waste incinerator sites.

Plant ______________________

Date ______________________

Location ______________________

Operator ______________________

Field Blank I.D.: Tenax ______________________

Tenax/Charcoal ______________________

Stack No. ______________________

Probe No. ______________________

VOST No. ______________________

Rotameter No. ______________________

Dry Gas Meter No. ______________________

Pair No.[b]	Leak Check	Cartridge I.D.		Rotameter Reading (1/min)	Time		Sampling Duration (min)	Probe Temp. (°C)	Barometric Pressure (in of Hg)	Condenser Outlet Temp. (°C)	Meter Temp. (Outlet)	Dry Gas Meter		Gas Sample Volume (liters)	Comments
		Tenax	Tenax/Charcoal		Initial	Final						Initial	Final		

Condensate Samples:

Run No. Sample I.D. Comments

NOTES: [a] Reading should be tabulated at the beginning of a run and every five minutes thereafter.

[b] Refers to sample collection on one pair of Tenax and Tenax/charcoal traps.

FIGURE 5. VOST FIELD DATA SHEET[a]

8.3.2 After leak checking but before the initiation of sample collection, the probe shall be purged with stack gas. This can be accomplished by attaching a pump to the isolation valve above the first condenser and drawing stack gas through the probe via the isolation valve, so that at the initiation of sample collection the probe is purged of ambient air.

8.4 Sample Collection

8.4.1 Prior to the initiation of sample collection, the probe shall be located in the stack at a point of average stack gas velocity and temperature. (These values can be determined using the procedures described in References 6 and 7.) Care should be exercised in the location of the probe in the stack to minimize effects of dilution air which may enter the stack through the sampling port and thereby impair the collection of a representative sample of the stack gas.

8.4.2 After leak checking (see Section 8.3.1), sample collection is accomplished by opening the valve at the inlet to the first condenser, turning on the pump, and sampling at a rate of 1 liter/minute for 20 minutes. The volume of sample for any pair of traps shall not exceed 20 liters. The temperature of the gas stream at the inlet to the first sorbent cartridge must be maintained at or below 20°C during sample collection (see Section 5.6.2).

8.4.3 After the collection of 20 liters of sample, the train is leak tested using the procedures described in Section 8.3. If this post leak test criterion is not met, the samples shall not be analyzed. If leak check criterion is met, the train is returned to atmospheric pressure as described in Section 8.3.1, the two sorbent cartridges removed, the end caps replaced, and the cartridges returned to a suitable container for storage and transport until analysis (see also Section 7.2.2).

8.4.4 In some cases, there may be a requirement to analyze the aqueous condensate. This situation may arise for water soluble volatile organic compounds. For these situations, preliminary laboratory evaluation of the sampling and recovery efficiency (including the purging efficiencies of the compounds) must be performed (Part B, Sections 7 and 11). Sampling and analysis procedures for water soluble volatile organics are currently being developed. In this case, when a pair of sorbent cartridges is changed, duplicate samples of the aqueous condensate should be poured into 40 ml glass vials equipped with plastic screw caps (Pierce 13075 or equivalent), and Teflon-faced silicon septa (Pierce 12722 or equivalent). If insufficient condensate is available, the vials shall be topped off with reagent water. The vials shall be carefully filled just to overflowing so that no air bubbles pass through the sample as the vial is being filled. The vial shall be sealed so that no air bubbles are entrapped in it. Vials shall be stored and transported at ice or cold pack temperature.

8.4.5 A new pair of cartridges is placed in the VOST. The VOST leak checked as described in Section 8.3 and the sample collection process repeated as described above. Sample collection continues until six pairs of cartridges have been taken (see Section 2.3). A maximum of six pairs of cartridges may be taken over a 2-hour sampling period (see Section 2.3).

8.4.6 All sample cartridges shall be stored on cold packs or ice until ready for analysis.

8.5 Field, Trip and Laboratory Blanks/Aqueous Field Blanks

8.5.1 Field Blanks - Blank Tenax and Tenax/charcoal cartridges are taken to the sampling site and the end caps removed for the period of time required (approximately 5 minutes) to exchange two pairs of cartridges on VOST. After the two VOST cartridges have been exchanged, the end caps are replaced on the blank Tenax and Tenax/charcoal tubes. These are returned to appropriate storage (see Section 7.2.2) and analyzed with the sample cartridges. At least one pair of field blanks (one Tenax, one Tenax/charcoal) shall be included with each six pairs of sample cartridges collected (or for each field trial using VOST to collect volatile POHCs). It is advisable to use two pairs of field blanks for each six pairs of cartridges collected.

8.5.2 Trip Blanks - At least one pair of blank cartridges (one Tenax, one Tenax/charcoal) shall be included with the shipment of cartridges to a hazardous waste incinerator site. These trip blanks will be treated as any other cartridges except that the end caps will not be removed during storage at the site. This pair of cartridges will be analyzed to monitor potential contamination which may occur during storage and shipment. One user (5) recommended including one pair of trip blanks for each VOST run conducted during a trial burn to obtain a more meaningful basis for blank correction of sample cartridges (Part B, Section 12.1.2).

8.5.3 Laboratory Blanks - One pair of blank cartridges (one Tenax, one Tenax/charcoal) will remain in the laboratory using the method of storage for field samples. If the field and trip blanks contain high concentrations of contaminants (e.g., greater than 2 ng of a particular POHC), the laboratory blank shall be analyzed in order to identify the source of contamination.

8.5.4 Aqueous Field Blanks - If the aqueous condensate is to be analyzed for volatile POHCs, an aqueous field blank is required. This consists of duplicate samples of reagent water placed in vials described in Section 8.4.3. These vials shall be stored and shipped in the same way as empty vials/aqueous condensate samples, and should be analyzed at the same time as the aqueous condensate samples. Additionally, a reagent water sample (prepared as above) will be retained in storage in the laboratory. If the analysis of aqueous field blanks shows the presence of POHCs, the corresponding laboratory blank shall be analyzed to verify that the residual contamination is not due to contamination of the aqueous field blanks at the time of preparation or during storage in the laboratory.

PART B

PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES FROM VOLATILE ORGANIC SAMPLING TRAIN

1. SCOPE AND APPLICABILITY

1.1 This method covers the analysis of volatile POHCs collected on Tenax and Tenax/charcoal sorbent cartridges using a VOST. Much of the description for purge-trap-desorb (P-T-D) GC/MS analysis is included in References 8 and 9. Since the majority of gas streams sampled using VOST will contain a high concentration of water, the analytical method is based on the quantitative thermal desorption of volatile POHCs from the Tenax and Tenax/charcoal cartridges and analysis by P-T-D GC/MS in order to minimize the effects of water on the analysis. This method is applicable to organic compounds with boiling points between 30° and 100°C. Many compounds with boiling points below 30°C and between 100° and 150°C, may be efficiently collected and analyzed using this method. However, laboratory evaluation of the collection and recovery efficiency for compounds with boiling points outside the range of 30° to 100°C (or those for which no VOST data are available) shall be performed (see Section 7).

1.2 This method is applicable to the analysis of Tenax and Tenax/charcoal cartridges used to collect volatile POHCs from wet stack gas effluents from hazardous waste incinerators.

1.3 The sensitivity of the analytical method for a particular volatile POHC depends on the level of interferences, the presence of detectable levels of volatile POHCs in blanks, and the ability to purge the volatile POHCs from water. The target detection limit for the method is 0.1 µg/m^3 (0.1 ng/l) in the stack gas. This corresponds to 2.0 ng adsorbed on a single pair of Tenax and Tenax/charcoal cartridges. Since 2 ng of a POHC may not be detectable by P-T-D GC/MS analysis, the method is designed to permit thermal desorption of up to five pairs of Tenax and Tenax/charcoal cartridges onto a single pair for analysis in order to achieve the target detection limit of the method. For a POHC present in the gaseous effluent at a concentration of 0.1 µg/m^3 (0.1 ng/l), the desorption of multiple pairs of cartridges onto a single pair for analysis can provide a total of 10 ng for analysis by P-T-D GC/MS.

1.4 This method is recommended for use only by experienced analytical chemists or under the close supervision of such qualified persons.

2. SUMMARY OF METHOD

2.1 A schematic diagram of the analytical system is shown in Figure 6. Pairs of the sorbent cartridges are spiked with an internal standard and thermally desorbed for 10 minutes at 180°C with organic-free nitrogen gas (at a flow rate of 100 ml/min), bubbled through 5 ml of organic-free water, and trapped on an analytical sorbent trap. After the 10-minute desorption, the

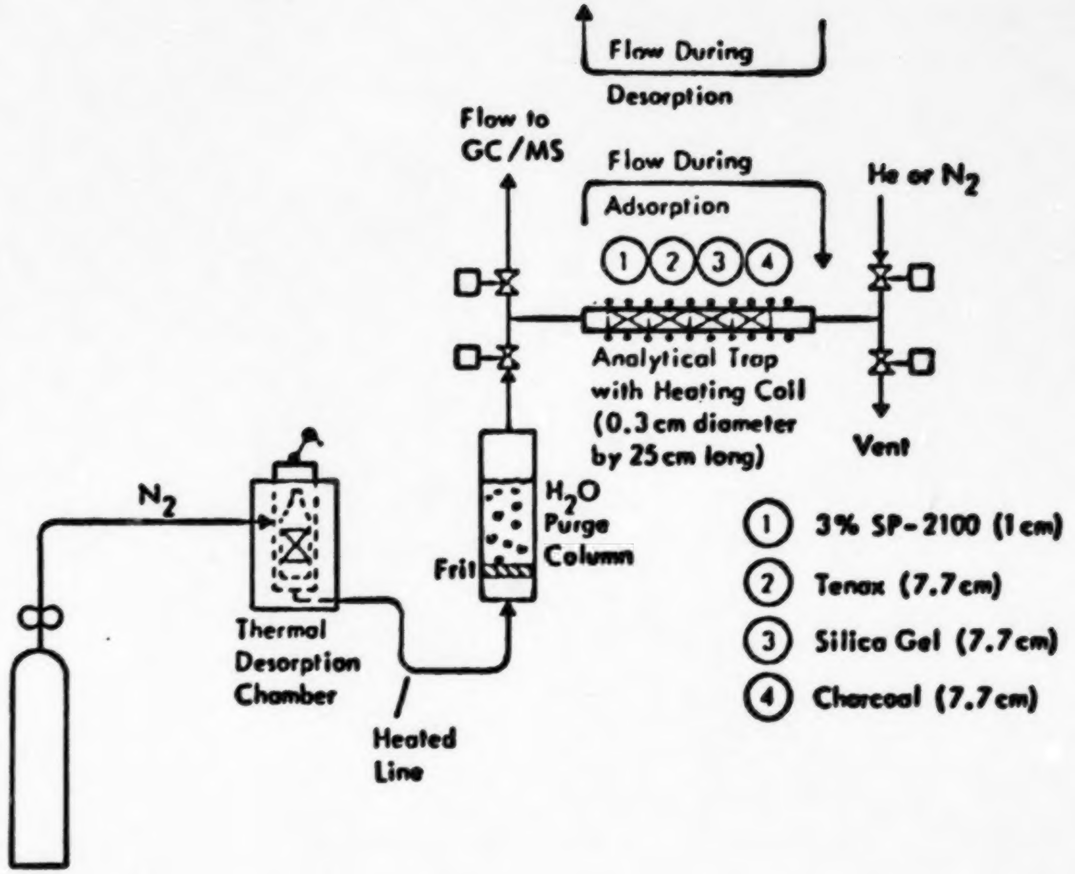

Flow During
Desorption
Flow to
GC/MS
Flow During
Adsorption
He or N_2
① ② ③ ④
Analytical Trap
with Heating Coil
(0.3 cm diameter
by 25 cm long)
Vent
N_2
H_2O
Purge
Column
Frit
Thermal
Desorption
Chamber
Heated
Line
① 3% SP-2100 (1 cm)
② Tenax (7.7 cm)
③ Silica Gel (7.7 cm)
④ Charcoal (7.7 cm)

FIGURE 6

SCHEMATIC DIAGRAM OF TRAP DESORPTION /ANALYSIS SYSTEM analytical sorbent trap is rapidly heated to 180°C with the carrier gas flow reversed so that the effluent flow from the analytical trap is directed into the GC/MS. The volatile POHCs are separated by temperature programmed gas chromatography and detected by low resolution mass spectrometry. The concentrations of volatile POHCs are calculated using the internal standard technique. To achieve increased sensitivity, multiple pairs of cartridges (up to five pairs) may be desorbed onto a single pair of Tenax and Tenax/charcoal cartridges. The resulting pair is analyzed by P-T-D GC/MS.

3. PRECISION AND ACCURACY

3.1 The overall accuracy of sample collection and analysis using VOST has been determined in laboratory evaluations to average ±50 percent for analysis of three replicate cartridges (5). These values may be revised as further evaluation of VOST performance is conducted.

3.2 The precision for the internal standard response (area) should be 20 percent during any given analysis period at a particular multiplier voltage of the mass spectrometer.

4. APPARATUS

4.1 Thermal Desorption Unit

4.1.1 The thermal desorption unit (Nutech Corporation Model 320 or equivalent) shall be capable of thermally desorbing the sorbent resin tubes. It shall be capable of rapidly heating the tubes to 180°C±10°C with flow of organic-free nitrogen through the tubes.

4.2 Purge and Trap Unit

4.2.1 The purge and trap unit consists of three separate pieces of equipment: the sample purger, analytical sorbent trap and the desorber. It should be capable of meeting all requirements of Sections 5.2 thru 5.2.4 of EPA Method 624 for analysis of purgeable organic compounds from water (8, 9). The analytical trap as specified in EPA Method 624 consists of 3% SP-2100, Tenax, silica gel, charcoal. Some users have found that improved performance can be achieved using only Tenax and charcoal in the analytical sorbent trap (5).

4.3 GC/MS System

4.3.1 Gas Chromatograph - An analytical system complete with a temperature programmable GC suitable for on-column injection, and all required accessories including syringes, analytical columns, and gases.

4.3.2 Column - Column dimensions shall be 1.8 m long by 0.25 cm I.D., stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent.

4.3.3 Mass Spectrometer - Capable of scanning from 20 to 260 amu every 7 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all criteria described in Method 624 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the GC inlet.

4.3.4 GC/MS Interface - Any GC to mass spectrometer interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest and achieves all performance criteria of Method 624 may be used. GC to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichloro-dimethylsilane.

4.3.5 Data System - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

5. REAGENTS

5.1 Reagent Water

5.1.1 Reagent water is defined as water in which an interferent is not observed at the detection limit of the parameters of interest.

5.1.2 Reagent water may be generated by passing tap water through a carbon filter bed containing about 450 grams of activated carbon (Calgon Corporation, Filtrasorb-300 or equivalent).

5.1.3 Reagent water may also be prepared by boiling distilled water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free, inert gas through the water for one hour. While still hot, the water should be transferred to a narrow mouth, screw cap bottle and sealed with a Teflon lined septum and cap.

5.1.4 Other methods which can be shown to produce reagent water can be used.

5.2 Analytical Trap Reagents

a) Tenax (60/80 mesh) - Chromatographic grade or equivalent

b) Methyl silicone packing - 3% OV-1 on Chromosorb W (60/80 mesh) or equivalent

c) Silica gel, Davison Chemical (35/00 mesh), Grade 15 or equivalent

d) Charcoal, petroleum based (SKC Lot 104 or equivalent) (see Section 6.2 in Part A)

5.3 Stock Standard Solution

5.3.1 Stock standard solutions will be prepared from pure standard materials or purchased certified solutions. The stock standards should be prepared in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

5.3.2 Fresh standards should be prepared weekly for volatile POHCs with boiling points of <30°C. All other standards must be replaced monthly, or sooner if comparison with check standards indicates a problem.

5.4 Secondary Dilution Standards

5.4.1 Using stock standard solutions, prepare secondary dilution standards in methanol which contain the compounds of interest either singly or mixed together, and at concentrations such that the desorbed calibration standards will bracket the working range of the analytical system.

5.5 Bromofluorobenzene (BFB) Standard

5.5.1 Prepare a 25 µg/ml solution of BFB in methanol.

5.6 Deuterated (d_6) Benzene Standard

5.6.1 Prepare a 25 µg/ml solution in methanol.

6. CALIBRATION

6.1 Assembly of P-T-D Device

6.1.1 Assemble a purge and trap desorption device (P-T-D) which meets all the requirements of Section 10.2 of this protocol and Section 5.2 of USEPA Method 624 (3).

6.1.2 Connect the thermal desorption device to the purge and trap device. Calibrate the P-T-D GC/MS system using the internal standard technique (Section 6.2).

6.2 Internal Standard Calibration Procedure

6.2.1 This approach requires the use of deuterated benzene as the internal standard for these analyses. Other internal standards may be proposed for use in certain situations. The important criteria for choosing a particular compound as an internal standard are that it be similar in analytical behavior to the compounds of interest, and it can be demonstrated that the measurement of the internal standard is not affected by method or matrix interferences. Other internal standards which have been used are d_{10}-ethylbenzene and d_4-1,2-dichloroethane. Several users add 50 ng of BFB to all sorbent cartridges (in addition to one or more internal standards) to provide continuous monitoring of the GC/MS performance relative to BFB.

6.2.2 Prepare calibration standards at a minimum of three concentration levels for each analyte of interest. The calibration standards are prepared by spiking a blank Tenax or Tenax/charcoal cartridge with a methanolic solution of the calibration standards (including 50 ng of the internal standard, such as deuterated benzene) using the flash evaporation technique.

6.2.3 The cartridge shall be mounted in the gas chromatograph so that the carrier gas enters the cartridge in the same direction as during sample collection (which is opposite to the direction of gas flow during P-T-D GC/MS analysis). The glass cartridge should be attached to the injection port of a gas chromatograph. The injector temperature is maintained at 160°C, and the carrier gas flow through the cartridges should be maintained at about 50 ml/min.

6.2.4 The flash evaporation technique requires filling the needle of a 5.0 (µl) syringe with clean methanol and drawing air into the syringe until the tip of the plunger reaches the 1.0 µl mark. This is followed by drawing a methanolic solution of the calibration standards (containing 25 µg/µl of the internal standard) until the tip of the plunger reaches the 3.0 µl mark. The contents of the syringe should be slowly expelled through the gas chromatograph injection port over about 15 seconds. After 25 seconds have elapsed, the gas flow through the cartridge should be shut off, the syringe removed, and the cartridge analyzed by the P-T-D GC/MS procedure outlined in Section 10.2. The total flow of gas through the cartridges during addition of calibration standard to blank cartridges, or internal standards to sample cartridges, should be 25 ml or less to avoid breakthrough of volatile sample components.

6.2.5 Analyze each calibration standard on both Tenax and Tenax/charcoal cartridges according to Section 10. The calibration procedures shall be identical to those used for analysis of sample cartridges. If cartridges are analyzed separately, then the Tenax and Tenax/charcoal cartridges must be calibrated separately. The direction of carrier gas flow through the cartridges during the desorption step should be opposite from the gas flow through the cartridge during sample collection so the inlet side of the cartridge during sample collection becomes the outlet side during P-T-D analysis. Tabulate the area response of the characteristic ions of each analyte against the concentration of the internal standard and calculate response factor (RF) for each compound using Equation 1.

$$RF = A_s C_{is} / A_{is} C_s \qquad [1]$$

where A_s = Area of the characteristic ion for the analyte to be measured

A_{is} = Area of the characteristic ion for the internal standard

C_{is} = Amount (ng) of the internal standard

C_s = Amount (ng) of the volatile POHC in calibration standard

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is} versus RF.

6.2.6 The working calibration curve or RF must be verified on each working day by the measurement of one or more of the calibration standards. If the response varies by more than ±25%, new calibration standards must be prepared and analyzed.

7. QUALITY CONTROL

7.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of blank Tenax and Tenax/charcoal cartridges spiked with the analytes of interest. The laboratory shall demonstrate collection and/or recovery efficiencies for compounds with boiling points outside the 30° to 100°C range, which meet the method criteria, prior to field use of the VOST (Part A, Section 3.2). The laboratory is required to maintain performance records to define the quality of data which are generated. Ongoing performance checks must be compared with established performance criteria to determine if results are within the expected precision and accuracy limits of the method. (Note: NBS traceable audit cylinders are being developed by Research Triangle Institute under the direction of USEPA-EMSL, Research Triangle Park, NC. The cylinders may be used for performance trials and evaluation of collection and recovery efficiencies in lieu of the methods specified in this section.)

7.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable precision and accuracy with this method. This ability is established as described in Section 7.2.

7.1.2 Recovery data for compounds boiling between 100° and 150°C must be provided which meets the method criteria (see Section 7.2.2). Laboratory evaluation is required in lieu of that data. The method of evaluation is that specified in Section 7.2 with the exception that it is not required to pull 20 liters of organic-free nitrogen through the sorbent cartridges.

7.1.3 Laboratory evaluation of collection and recovery efficiencies shall be performed for compounds with boiling points below 30°C. Due to the possibility of sample breakthrough and poor recovery of the sample from the charcoal, the evaluation shall be comparable to that described in Reference 5. A gas stream containing the POHC shall be generated and sampled using the VOST. The sampling rate for the laboratory evaluation shall be the same as that proposed for field application. The laboratory evaluation must meet the precision and accuracy criteria described in Section 7.2.2.

7.1.4 The laboratory must spike all Tenax and Tenax/charcoal cartridges with the internal standard(s) to monitor continuing laboratory performance. This procedure is described in Section 6.2.

7.2 To establish the ability to generate acceptable accuracy and precision, the analyst must spike blank Tenax and Tenax/charcoal with the analytes of interest at two concentrations in the working range by the flash vaporization technique discussed in Sections 6.2.3 and 6.2.4. The cartridges are removed from the gas chromatograph and connected to a source of organic-free nitrogen. The cartridges sample 20 liters of nitrogen at a flow rate equivalent to that proposed for field use of VOST.

7.2.1 The average response factor (RF) and the standard deviation (S) for each must be calculated.

7.2.2 The average recovery and standard deviation must fall within the expected range for determination of volatile POHCs using this method. The expected range for recovery (indicative of accuracy) of volatile POHCs using this method is ±50 percent. The expected standard deviation is 25 percent.

7.3 The analyst must calculate method performance criteria for the internal standard.

7.3.1 Calculate upper and lower control limits for method performances using the average area response (A) and standard deviation (S) for internal standard:

Upper Control Limit (UCL) = A + 3S

Lower Control Limit (LCL) = A - 3S

The UCL and LCL can be used to construct control charts which are useful in observing trends in performance. The control limits must be replaced by method performance criteria as they become available from the USEPA.

7.4 Each day, the analyst must demonstrate through analysis of blank Tenax and Tenax/charcoal cartridges and reagent water that interferences from the analytical system are under control.

8. SAMPLE COLLECTION, PRESERVATION AND HANDLING

8.1 The requirements for sample collection, preservation, and handling are described in Part A of this protocol.

9. DAILY GC/MS PERFORMANCE TESTS

9.1 The daily GC/MS performance tests required for this method are described in Section 10 of EPA Method 624 (9). The key abundance criteria for BFB which must be met before any samples are analyzed are listed below. This can be done by injecting 50 ng of BFB directly on the column or by adding 50 ng of BFB to a blank Tenax sorbent cartridge (see Sections 6.2.3 and 6.2.4) and desorbing this cartridge according to the procedures described in Section 10. The latter procedure is preferred when all sample sorbent cartridges are spiked with BFB in addition to one or more of the internal standards discussed in Section 6.2.1.

BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

10. SAMPLE DESORPTION AND GAS CHROMATOGRAPHY

10.1 The P-T-D GC/MS procedures are those described in Section 11 of Method 624 (9) with the addition of the procedure described in Section 10.2 for desorption of Tenax and Tenax/charcoal cartridges.

10.2 The schematic of the P-T-D GC/MS system is shown in Figure 6. The sample cartridge is placed in the thermal desorption apparatus (Nutech 320 or equivalent) and desorbed in the P-T-D system by heating to 180°C for 10 minutes. Sample cartridges may be desorbed in pairs. However, if the analyte concentrations are anticipated to be sufficiently high to saturate the GC/MS when desorbed in pairs, consideration should be given to individual analysis of cartridges. The desorbed components pass into the bottom of the water column, are purged from the water and collected on the analytical sorbent trap. After the 10-minute desorption period, the compounds are desorbed from the analytical sorbent trap into the GC/MS system according to the procedures described in Section 11 of Method 624.

11. AQUEOUS CONDENSATE

11.1 Analysis of aqueous condensate samples should be performed according to the procedures described in References 8 and 9. If the volatile POHCs of interest are water soluble, it is necessary to demonstrate that this method is capable of recovery of the volatile POHCs of interest using Method 624. This can be accomplished by P-T-D GC/MS analysis of reagent water which has been fortified with the analytes of interest.

11.2 If adequate recovery of water soluble volatile POHCs cannot be achieved using the procedures described in References 8 and 9, alternative methods for analysis, such as direct aqueous injection, may be required. If an alternative analytical method is used, method performance must be documented by analysis of laboratory reagent water which has been fortified with the volatile POHCs of interest.

11.3 Methods for analysis of water soluble volatile POHCs in aqueous condensate are currently under development.

12. QUALITATIVE IDENTIFICATION

12.1 The qualitative identification procedure of volatile POHCs using this protocol is described in Section 12 of Method 624 (9).

13. CALCULATIONS

13.1 When an analyte has been qualitatively identified, quantification should be based on the integrated abundance from the EICP of the primary characteristic ion chosen for that analyte. If the sample produces an interference for the primary characteristic ion, a secondary characteristic ion should be used.

13.1.1 Using the internal standard calibration procedure, the amount of analyte in the sample cartridge is calculated using the response factor (RF) determined in Section 6.2.5 and Equation 2.

$$\text{Amount of POHC} = A_sC_{is}/A_{is}RF \qquad [2]$$

where A_s = Area of the characteristic ion for the analyte to be measured

A_{is} = Area for the characteristic ion of the internal standard

C_{is} = Amount (ng) of internal standard

13.1.2 The choice of methods for evaluating data collected using VOST for incinerator trial burns is a regulatory decision. The procedures used extensively by one user are outlined below (10).

13.1.3 The total amount of the POHCs of interest collected on a pair of traps should be summed. These values should then be blank corrected. Guidelines for blank correction of sample cartridges are outlined below.

13.1.3.1 After all blanks (field and trip) are analyzed, a paired t-test should be used to determine whether trip blanks are significantly different from field blanks. If no difference is found, then the mean and standard deviation of the combined field and trip blanks for each POHC of interest is calculated, and that value is used as the blank.

13.1.3.2 If, using the paired t-test, the field and trip blanks are determined to be different, then the field blank (or the mean of multiple field blanks) associated with a particular run should be used as the blank value for that particular run.

13.1.3.3 If an individual field blank is used as the blank value for a particular run, the VOST user must evaluate whether this blank is different from sample cartridges associated with this blank. Although no specific criteria are offered here for guidance in this case, specific criteria for use of individual field blanks for blank correction will be developed as more data for use of VOST becomes available. This situation can be alleviated by including two field blanks (i.e., two blank Tenax and two blank Tenax/charcoal cartridges) per run.

13.1.4 Next, for each sample/POHC combination, a determination must be made as to whether a particular sample is significantly different from the associated blank. If the mean of the trip and field blanks is used, then a sample is different from the blank if:

(measured sample value)-(mean blank value) ≥ 3

x blank standard deviation

If the sample is determined to be different from the blank according to the above criteria, then the emission value of a particular POHC is blank corrected by subtracting the mean blank value (as defined in Section 12.1.3) from the measured sample value. (If an individual field blank is used as the blank value, the above criteria do not apply.)

13.1.5 If, according to the above procedures, the sample cannot be distinguished from the blank (i.e., for a given POHC there is a high sample value and high blank value or there is a low sample value and low blank value), the measured sample value is not blank corrected. In this case, the measured sample value is used to calculate a maximum emission value (and therefore a minimum DRE value) for that particular run.

13.1.6 The observation of high concentrations of POHCs of interest in blank cartridges indicates possible residual contamination of the sorbent cartridges during shipment and use at the site. Data which fall in this category (especially data indicating high concentrations of POHCs in blank sorbent cartridges) should be qualified with regard to validity, and blank data should be reported separately. The applicability of data of this type to the determination of DRE is a regulatory decision. Continued observation of high concentrations of POHCs in blank sorbent cartridges indicates that procedures for cleanup, monitoring, shipment and storage of sorbent cartridges by a particular user, be investigated to eliminate this problem.

13.1.7 After blank corrections have been made, the amounts of individual POHCs on each sorbent cartridge used for a given run is summed and divided by the volume of gas sampled to obtain the concentration of each POHC in the stack gas.

13.1.8 If any internal standard recoveries fall outside the control limits established in Section 7.3.1, data for all analytes determined for that cartridge(s) must be qualified with the observation.

14. METHOD PERFORMANCE

14.1 The method detection limit, average recoveries and standard deviation of the average recoveries of the analytes determined using this method have not yet been established. The method performance will be documented as more data become available.

REFERENCES

1) Resource Conservation and Recovery Act, Subtitle [3001-3013,42 USC 6921-6934 (1976) and Supplement IV (1980)].

2) Vogel, G., et al. "Guidance Manual for Hazardous Waste Incineration Permits," EPA-68-01-0092, SW-966, NTIS PB-84/100577, July 1982.

3) Code of Federal Regulations, Title 40, Part 60, Appendix A, 1980.

4) Harris, J. C., et al. "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA 600/8-84-002, February 1984.

5) Jungclaus, G., et al. "Development of a Volatile Organic Sampling Train (VOST)," Presented at 9th Annual Research Symposium, Land Disposal, Incineration and Treatment of Hazardous Wastes, Ft. Mitchell, KY, May 2-4, 1983.

6) Federal Register, 42, 41758-41768, August 18, 1977.

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8) Federal Register, 44, 69464, December 3, 1979.

9) Longbottom, J. and J. Lichtenberg, eds., "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater; Test Methods," Method 624, EPA-600/4-82-057, 1982.

10) Trenholm, A. Midwest Research Institute, Personal Communication, August 5, 1983.

APPENDIX G

DILUTION SAMPLING FOR CONDENSIBLE MATERIAL

TASK FOR SOUTHERN RESEARCH INSTITUTE TO OPERATE STACK DILUTION SAMPLING SYSTEM

Technical Directive 135 - Dilution Sampling for Condensible Material

Introduction

The test data from the AFBC-Hazardous Waste Incineration test conducted at the beginning of May has been compiled, with the exception of the chemical analysis of the SDSS filters and rinse samples. The GC/MS analysis of the samples, which were turned over to EPA personnel, has not been completed. A partial test report concerning Southern Research's involvement was sent to Ray Merrill for inclusion in the EPA internal final report on the test burn. The following is a preliminary report concerning all phases of the SDSS test with the exception of the sample analyses.

Combustion Facility Background

The atmospheric fluidized bed combustion facility (AFBC), located at EPA's National Environmental Research Laboratory in Research Triangle Park, North Carolina is the site of a three phase program designed to establish a fully permitted hazardous waste research facility. During the first two weeks of May 1983, Phase I of this program was completed with a test burn of the AFBC. The objectives of Phase I include (1) development, testing, and refinement of incinerator operating protocol, (2) establishing destruction and removal efficiencies (DRE) of the AFBC for selected surrogate wastes, and (3) the testing of selected state-of-the-art sampling and analysis trains.

The combustor was previously used for coal combustion research; therefore, a few minor modifications were necessary before Phase I testing could begin. Damage to the old refractory dictated the need to recast the combustion section. A high abrasion resistant material (Babcock and Wilcox Kao-Phos 93) was used. It was also known to have a high chemical resistance to chlorine, a major component of future studies. A gas preheater and liquid fuel feed system were also added to the AFBC. The gas preheater was installed to provide a more dependable and quicker performance and as an ignition source for the liquid fuels. The liquid fuel system is required to fire the AFBC on No. 2 fuel oil and waste oil mixture.

The sampling methods used were (1) the Source Assessment Sampling System (SASS) with a glass module assembly for measuring semi-volatile organic compounds, (2) the Volatile Organic Sampling Train (VOST), and (3) the Stack Dilution Sampling System (SDSS) for "non-volatile" (condensible) detection. The glass SASS and the SDSS were tested simultaneously, while the VOST was scheduled to be used following the first tests.

Two waste/fuel mixtures were used. One contained volatile compounds for the VOST, and the other contained semi-volatile and condensible compounds for testing with the remaining two trains. Since this task concerns only the

SDSS, it will only address the condensible compounds. Initially, three condensible compounds were to be used: triphenylmethane, triphenylene, and 2,4,6-triphenoxy-S-triazine. These compounds were selected for a variety of reasons with safety being the primary concern. However, due to cost constraints and difficulties in solubility, the only condensible compound used during the testing was triphenylmethane.

The operating protocol of the AFBC facility used for the surrogate waste incineration tests is similar to most oil-fired combustion facilities, with the exception of the waste oil addition. The air is turned on to the system prior to the igniting of the gas-fired preheater. As the bed is heating up, the main air valve is adjusted to maintain the desired fluidization velocity. When the bed reaches approximately 1000°F, the clean fuel oil is slowly introduced. The gas flow to the preheater is cut off, but purge air is maintained to cool the nozzle. When the bed temperature reaches about 1850°F, the fuel feed is reduced to maintain desired temperatures and velocities. When steady state is reached the clean oil is replaced by the fuel/waste mixture. It should be noted that fuel flow adjustments are needed to compensate for the difference in BTU value of the spiked fuel. At the end of the test period the fuel/waste mixture is replaced with clean oil to flush out the feed lines. As the combustor cools down the air preheater must be restarted to insure a slow cool down period. The preheater is shut off when the bed reaches approximately 800°F, and the blower and draft fan are shut off when the bed temperature drops below 600°F.

Test Conditions

The Stack Dilution Sampling System was brought on site May 3, 1983 by Ashley Williamson and Dicky Armstrong, the sampling team from Southern Research. The first three days were spent attending mandatory safety classes and setting up the sampling train. One test, HAZ1 (4 hours), was completed on Thursday, May 5. On May 9, Randy Martin replaced Dicky Armstrong and completed the final two tests, HAZ2 (5 1/2 hours) and HAZ3 (5 1/2 hours), on May 10 and May 12, respectively.

As can be seen in Figure 1, the SDSS sampling location was immediately upstream of the flue gas cooler. This presented problems for isokinetic sampling. Initially, it was thought the flowrate of the flue gas would be in the range of 15-30 scfm at a stack temperature between 500-600°F. In order to isokinetically sample at the desired flowrate a special buttonhook nozzle was machined. However, upon arriving at the test site it was learned the flue gas flowrate would be around 60 scfm and 1200°F at the sampling location. This allowed the use of a standard buttonhook 1/2" nozzle. The higher temperature, however, posed a threat to the teflon components of the SDSS. Therefore, a 3 foot length of 1/2" stainless steel tube was used as an uninsulated probe extension between the instack nozzle and the probe. The tube sufficiently cooled the gas before reaching the probe which was heated to 150°C to prevent premature condensation of the sample. Typical gas temperature at the entry of the heated teflon hose were 170°C.

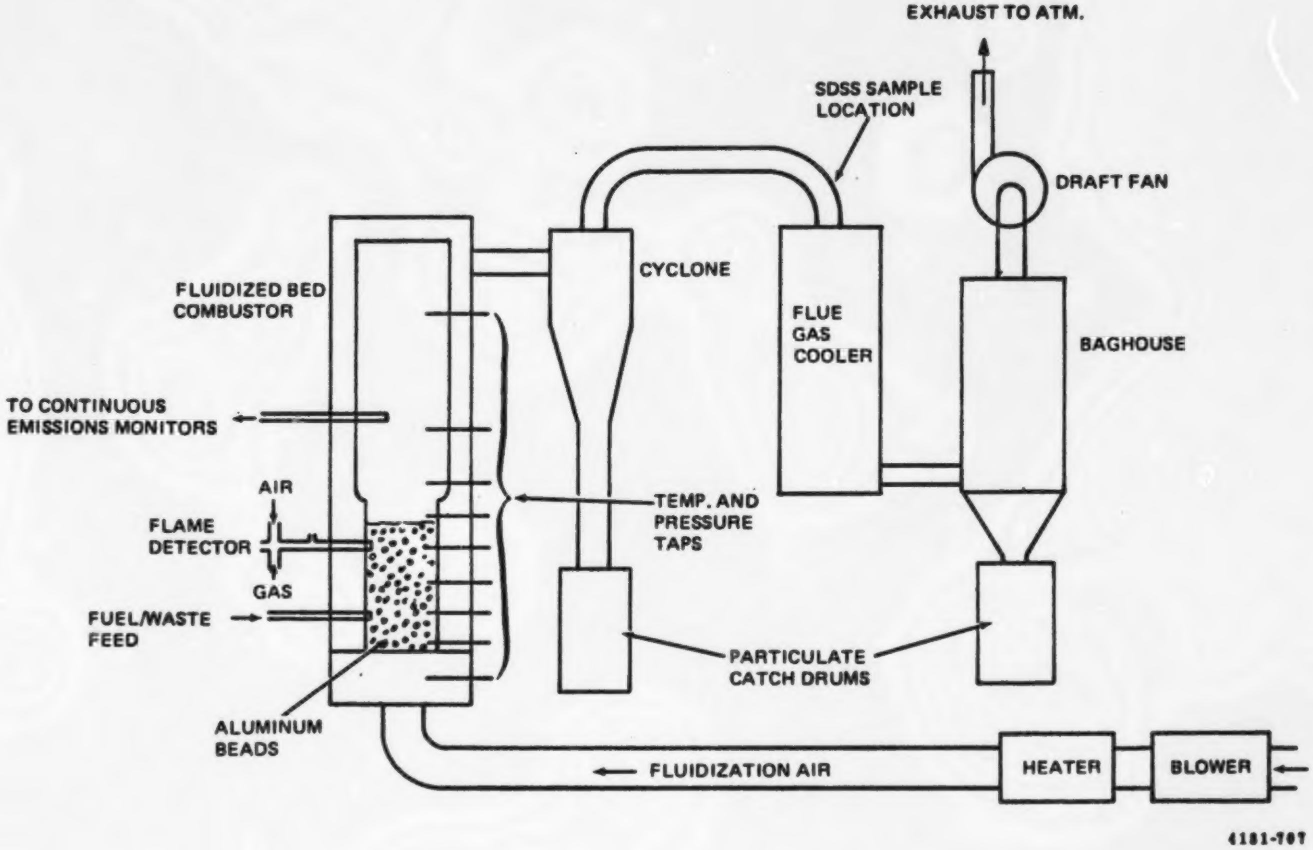
EXHAUST TO ATM.
SDSS SAMPLE LOCATION
DRAFT FAN
FLUIDIZED BED COMBUSTOR
CYCLONE
FLUE GAS COOLER
BAGHOUSE
TO CONTINUOUS EMISSIONS MONITORS
AIR
FLAME DETECTOR
GAS
FUEL/WASTE FEED
TEMP. AND PRESSURE TAPS
PARTICULATE CATCH DRUMS
ALUMINUM BEADS
FLUIDIZATION AIR
HEATER
BLOWER
4181-707

Figure 1. Phase I facility schematic.

Table 1. Sampling Data from AFBC-Hazardous Waste Incineration Test

	HAZ1	HAZ2	HAZ3
Avg. Stack Temperature (°K)	955.3	905.3	902.0
% CO_2	6.1	6.1	5.8
% O_2	12.8	12.8	12.6
CO, ppm	-	-	17
% Excess Air	148.7	148.7	140.9
% H_2O	4.5	4.6	4.5
Sample Time, minutes	238	320	326
Sample Flowrate, acfm	1.7663	2.5693	2.6019
Target Flowrate, acfm	1.7681	2.6271	2.9928
% Isokinetic	99.9	97.8	86.9
Duct Pressure (in. Hg absolute)	29.54	30.08	29.86
Duct Velocity, ft/sec	21.613	32.113	36.581
Sample Volume, $dsft^3$	121.659	255.360	262.635
Sample Volume, dnm^3	3.445	7.231	7.437

Under normal operating procedures, the SDSS operates using an IP Dual Cyclone Sampler, in which particles with an aerodynamic diameter greater than 15 μm and those in the range 2.5-15 μm are removed in two stages. The three inch port on the duct, however, prohibited the use of the dual cyclone train. Deletion of the cyclone train made isokinetic sampling easier.

Prior to the first test run, the entire SDSS train was rinsed with dichloromethane to insure a chemically clean background for the tests. The rinse solvent samples were placed in individual, foil-wrapped glass bottles uniquely marked identifying the system location of the rinse. These blank rinses were then released to Ray Merrill of TSS/IERL for analysis.

Table 1 contains the run data for the SDSS train for the three test burns. After each sampling run, the probe was immediately removed from the duct and all the heat to the system was turned off. The preweighed 8 x 10 teflon filter was removed from the dilution chamber, folded in quarters (loaded side in), and placed in a foil-covered, uniquely-labelled glass bottle. A small vial containing several crystals of silica gel was placed in the bottle with the filter. After a dessication period of 24 hours the filter was reweighed to determine the mass loading.

The sample lines were rinsed with dichloromethane (Burdick & Jackson) and the total rinse from a test run was placed in foil-covered glass bottle. Previous laboratory studies showed little sample collection in the dilution chamber itself; therefore, the chamber was not rinsed. All other parts of the SDSS were rinsed twice sequentially with dichloromethane from a teflon rinse bottle according to the following protocol:

- Nozzle: 2 x 25 ml - wipe tip, rinse away from tip; add to probe wash.
- Probe: 2 x 100 ml - cap ends, rotate and slosh back and forth.
- Hose: 2 x 100 ml - cap ends, rotate and slosh back and forth.
- Inlet: 2 x 50 ml - cap bottom and venturi pressure taps, squirt from top.

The samples (filters and rinses) were then turned over to Ray Merrill of TSS/IERL for GC/MS analysis. A sample of 200 ml of the dichloromethane was also delivered to EPA personnel to be used as a solvent blank.

Results and Conclusions

The raw weights and particulate concentration as measured by the SDSS are shown in Table 2. The masses collected from the rinses are not included because the rinses were turned over to EPA personnel for chemical analysis. Under normal test procedure the rinses would be transferred into tared weighing boats and the solvent would be evaporated. The mass of the particulate collected during the rinse would then be included in the total system mass

Table 2. Raw Weights and Particulate Concentration on SDSS Filter From AFBC - Hazardous Waste Incineration Test

Run	Raw Weight (mg)	Particulate Concentration (mg/dnm^3)
HAZ1	46.1	13.4
HAZ2	174.0	24.1
HAZ3	204.3	27.5

collected. However, since the rinse visually appeared clear, little mass is expected from the rinses.

The final report concerning the entire task, including both laboratory and field test work, will be written when the chemical analyses of the samples are complete.